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Document number: SD-WM-DP-145

Section 5 **of** 12

Title: REVISED 60 DAY SAFETY SCREENING + FERROCYANIDE RESULTS FOR TANK 241-BY-108 ROTARY SAMPLES CORE 98 + CORE 104

Date: 02/02/96 **Revision:** R001

Originator: BALDWIN JH
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References: ECN-629001

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PART III

WHC-SD-WM-DP-145, REV. 1

SINGLE SHELL TANK WASTE CHARACTERIZATION FOR TANK 241-BY-108 PERFORMED AND REPORTED TO WESTINGHOUSE HANFORD BY PACIFIC NORTHWEST LABORATORY

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WHC-SD-WM-DP-145, REV. 1

SINGLE SHELL TANK

WASTE CHARACTERIZATION

CORE 99

Tank BY-108

Revision 1

September 29, 1995

Prepared by: KL Silvers
LR Greenwood
RT Steele
JM Tingey
MW Urie

Pacific Northwest Laboratory

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This Data Package contains the results obtained by the Pacific Northwest Laboratory (PNL) 325 Analytical Chemistry Laboratory (ACL) and 325 High Level Radiochemistry Facility (HLRF) staff for the characterization and analyses of Core 99 taken from Single-Shell Tank (SST) BY-108. The characterization and analysis requirements for Tank BY-108 are outlined in the "Fiscal Year 1995 Tank Waste Remediation System Tank Waste Analysis Plan," (WHC-SD-WM-PLN-091, Revision 0). Specific characterization activities are detailed in the "Tank 241-BY-108 Tank Characterization Plan," (WHC-SD-WM-TP-275, Revision 0E).

As stated in the TCP, the work performed on Tank BY-108 represents an opportunistic venture; the results are to be used to gain further information/ insight on the nature of Tank BY-108.

The Tank Characterization Plan (TCP) prescribes the analytical requirements in the form of selected methods, analytes of interest, type of preparation, quality control, reporting units and notification limits. The TCP also establishes the analytical priority and provides decision points for the analytical laboratory on whether or not to proceed with additional analysis. The decision points are communicated by "if - then" type criteria. These decision points are administered as follows. If an analytical measurement exceeds a set notification threshold, then the lab proceeds with the additional analyses. This approach, although an effective means of eliminating analytical delays, requires that analyses be performed in series, thus requiring a defined amount of time. In the case of Tank BY-108 sample analyses, 35 calendar days were allotted to complete the entire scope of work. This relatively short analytical window precluded the effective utilization of the "if - then" decision strategy. As a result, a number of managerial decisions were made in the planning stage to perform additional analyses in advance of exceeding notification limits.

In Table C-4 of the TCP the analytes of interest were not identified for ion chromatography (IC) or inductively coupled plasma (ICP). For these methods, the data quality objectives documents were reviewed and analytes of interest identified. The analytes of interest for the IC include nitrate, nitrite, sulfate and phosphate. The analytes of interest identified for the ICP were iron, aluminum, manganese, nickel, bismuth and sodium. These analytes of interest were evaluated against precision and accuracy requirements in the TCP. Two QC approaches were utilized to acquire the accuracy information; serial dilutions were used when the analyte concentration was expected to be high and matrix spikes were used when the analyte concentration was expected to be relatively low or unknown. Although the analytes of interest were the primary focus of each analysis, additional analytes/results were available and are provided for informational purposes.

Specific analyses for each segment and quarter segment taken from Core 99 are described in Test Instructions prepared by the PNL Project Manager in accordance with administrative procedures contained in the Analytical Chemistry Laboratory Procedure Compendium (PNL-MA-599).

Core 99 contained four segments. Each segment was extruded and sub-sampled into segments and quarter segments at the WHC 222-S Laboratory. The PNL HLRF received the last shipment of BY-108 segments on August 25, 1995. All of the samples were received without chain-of-custody documentation. During the extrusion and sub-sampling of each segment at 222-S, a separate aliquot of sample was removed for thermogravimetric analysis. These aliquots were placed in separate uniquely identified containers and shipped with the remaining sample material. All extruded sample material from Core 99 Segments 1 through 3 were transferred to PNL. Segment 4 was further subsampled and approximately 30 g of each quarter segment was shipped to PNL for analyses. The number of sample containers received, the approximate sample weights and volumes are as follows for Core 99, Segments 1-4. Segment 1 consisted of two sample containers of solids with a combined weight of 56 g. Segment 2 consisted of four sample containers, three had solids with a combined weight of 37 g while one had 230 mL of drainable liquid. Segment 3 consisted of three sample containers, two had solids with a combined weight of 22 g while one had 25 mL of drainable liquid. Segment 4 consisted of eight sample containers of solids with a combined weight of 118 g. Table 1-1 provides a with a combined weight of 118 g. Table 1-1 provides a more detailed breakdown of sample identification and approximate sample volumes received from WHC.

All Core 99 sample material was homogenized prior to analysis. Segment 1 and Quarter Segment 4B were homogenized and sub-sampled from the top and bottom. Each sub-sample was analyzed in duplicate by ICP and GEA to test the homogenization methodology. The results of the homogenization test are presented in Table 1-2. The small volume of solid sample material received for each quarter segment made it difficult to thoroughly homogenize the samples. The relatively non-homogenous sample material, in conjunction with the small quantities of sample used in the preparations, increased the likelihood of having high relative percent differences (RPDs) between samples and associated duplicates. In some instances, the TCP precision requirement of 90-110% could not be met.

The ACL sample numbers assigned to the segment and quarter segment samples and the associated analyses are listed in Tables I-1 through I-4. The data within this package are divided into three groups: Physical Testing, Inorganic Analysis and Radiochemical Analysis. All chemical analysis data are reported on a wet-weight basis. That is, no corrections have been made for the water content in the samples. The analytical results for the drainable liquids were converted to a g/mL basis using a density of 1.43 g/mL. The density was calculated during the sample preparation (i.e., water leach). All required sample preparations were performed in duplicate. The quality control (QC) requirements for each sample are defined in the Test Instructions. Samples were prepared and analyzed as batches where feasible. A minimum number of QC samples were analyzed in each batch and all QC data are included in this data package.

In several instances, the precision and accuracy identified in the TCP could not be met. This fact is clearly identified and discussed in the following sections. The limited quantity and complex nature of the material tested and the small quantities taken for preparation negatively impact the laboratory's ability to meet the TCP criteria. The TCP criteria appears to be unattainable with any consistency for this matrix.

Table I-1: Tank BY-108, Core 99, Segment 1

Analysis	Segment 1 Unhomogenized	Segment 1	
TGA	95-07931		Run in Duplicate
Water Leach		95-07941-C1	Water Leach Sample (IC)
		95-07941-C2	Water Leach Duplicate
		95-07941-C3	Methods Blank (one per batch)
		95-07941-C4	Spike (IC Only) (one per batch)
		95-07941-C5	Blank Spike (IC Only) (one per batch)
Direct		95-07941-D1	DSC & Density Sample
		95-07941-D2	DSC & Density Duplicate
		95-07941-D3	Methods Blank (one per batch)
Total Cyanide		95-07941-G1	Total CN Sample
		95-07941-G2	Total CN Duplicate
		95-07941-G3	Methods Blank (one per batch)
		95-07941-G4	Matrix Spike (one per batch)
		95-07941-G5	Blank Spike (one per batch)
Homogenization Test Fusion Dissolution KOH		95-07941-H1T	Homogenization Test Fusion Sample - Top *
		95-07941-H2T	Homogenization Test Fusion Duplicate - Top
		95-07941-HCB	Homogenization Test Fusion Blank
		95-07941-H1B	Homogenization Test Fusion Sample - Bottom
		95-07941-H2B	Homogenization Test Fusion Duplicate - Bottom
Fusion Dissolution KOH		95-07941-H1	KOH Fusion Sample (ICP, Radchem**)
		95-07941-H2	KOH Fusion Duplicate
		95-07941-H3	Methods Blank (one per batch)
		95-07941-H5	Post Digestion Blank Spike (ICP only)
		95-07941-H6	Post Digestion Spike (ICP Only)
Carbon		95-07941-J1	Carbon Analysis Sample (TIC/TOC/TC)
		95-07941-J2	Carbon Analysis Duplicate
		95-07941-J3	Methods Blank (one per batch)
		95-07941-J4	Matrix Spike (one per batch)
Fusion Dissolution Na2O2		95-07941-N1	Na2O2 Fusion Sample (ICP)
		95-07941-N2	Na2O2 Fusion Duplicate (one per batch)
		95-07941-N3	Methods Blank (one per batch)
		95-07941-N5	Post Digestion Blank Spike (ICP only)
		95-07941-N6	Post Digestion Spike

* Homogenization Test: ICP & GEA

** Fusion Radchem: Total Alpha, Total Beta, Sr-90, PU-239/240, GEA, U)

Table 1-2: Tank BY-108, Core 99, Segment 2

Analysis	Segment 2 Drainable Liquid	Quarter Segment 2A Unhomogenized	Quarter Segment 2D Unhomogenized	Quarter Segment 2A	Quarter Segment 2D	
TGA	95-07932	95-07933	95-07934			Run in Duplicate
Acid Digestion	95-07932-A1					Acid Digestion Sample (ICP, Radchem *)
	95-07932-A2					Acid Digestion Duplicate
	95-07932-A3					Methods Blank (one per batch)
	95-07932-A4					Matrix Spike (one per batch)
	95-07932-A5					Blank Spike (one per batch)
Water Leach	95-07932-C1			95-07942-C1	95-07943-C1	Water Leach Sample **
	95-07932-C2			95-07942-C2	95-07943-C2	Water Leach Duplicate
	95-07932-C3			95-07942-C3	95-07943-C3	Methods Blank (one per batch)
	95-07932-C4			95-07942-C4	95-07943-C4	Spike (IC Only) (one per batch)
	95-07932-C5			95-07942-C5	95-07943-C5	Blank Spike (IC Only) (one per batch)
Direct	95-07932-D1			95-07942-D1	95-07943-D1	Direct Sample ***
	95-07932-D2			95-07942-D2	95-07943-D2	Direct Duplicate
	95-07932-D3			95-07942-D3	95-07943-D3	Methods Blank (one per batch)
Total Cyanide	95-07932-G1			95-07942-G1	95-07943-G1	Total CN Sample
	95-07932-G2			95-07942-G2	95-07943-G2	Total CN Duplicate
	95-07932-G3			95-07942-G3	95-07943-G3	Methods Blank (one per batch)
	95-07932-G4			95-07942-G4	95-07943-G4	Matrix Spike (one per batch)
	95-07932-G5			95-07942-G5	95-07943-G5	Blank Spike (one per batch)
Fusion Dissolution KOH				95-07942-H1	95-07943-H1	KOH Fusion Sample (ICP, Radchem****)
				95-07942-H2	95-07943-H2	KOH Fusion Duplicate
				95-07942-H3	95-07943-H3	Methods Blank (one per batch)
				95-07942-H5	95-07943-H5	Post Digestion Blank Spike (ICP only)
				95-07942-H6	95-07943-H6	Post Digestion Spike (ICP Only)
Carbon	95-07932-J1			95-07942-J1	95-07943-J1	Carbon Analysis Sample (TIC/TOC/TC)
	95-07932-J2			95-07942-J2	95-07943-J2	Carbon Analysis Duplicate
	95-07932-J3			95-07942-J3	95-07943-J3	Methods Blank (one per batch)
	95-07932-J4			95-07942-J4	95-07943-J4	Matrix Spike (one per batch)
Fusion Dissolution Na2O2				95-07942-N1	95-07943-N1	Na2O2 Fusion Sample (ICP)
				95-07942-N2	95-07943-N2	Na2O2 Fusion Duplicate (one per batch)
				95-07942-N3	95-07943-N3	Methods Blank (one per batch)
				95-07942-N5	95-07943-N5	Post Digestion Blank Spike (ICP only)
				95-07942-N6	95-07943-N6	Post Digestion Spike

* Acid Digestion Radchem: Total Alpha, Total Beta, GEA
** Water Leach: IC on Drainable Liquid and Quarter Segments
*** Direct Sample: DSC on Drainable Liquid, DSC and Density on Quarter Segments
**** Fusion Radchem: Total Alpha, Total Beta, Sr-90, PU-239/240, GEA, U)

Table I-3: Tank BY-108, Core 99, Segment 3

Analysis	Segment 3 Drainable Liquid	Quarter Segment 3A Unhomogenized	Quarter Segment 3A	
TGA	95-07935	95-07936		Run in Duplicate
Acid Digestion	95-07935-A1			Acid Digestion Sample (ICP, Radchem *)
	95-07935-A2			Acid Digestion Duplicate
	95-07935-A3			Methods Blank (one per batch)
	95-07935-A4			Spike (one per batch)
	95-07935-A5			Blank Spike (one per batch)
Water Leach	95-07935-C1		95-07944-C1	Water Leach Sample **
	95-07935-C2		95-07944-C2	Water Leach Duplicate
	95-07935-C3		95-07944-C3	Methods Blank (one per batch)
	95-07935-C4		95-07944-C4	Spike (IC Only) (one per batch)
	95-07935-C5		95-07944-C5	Blank Spike (IC Only) (one per batch)
Direct	95-07935-D1		95-07944-D1	Direct Sample ***
	95-07935-D2		95-07944-D2	Direct Duplicate
	95-07935-D3		95-07944-D3	Methods Blank (one per batch)
Total Cyanide	95-07935-G1		95-07944-G1	Total CN Sample
	95-07935-G2		95-07944-G2	Total CN Duplicate
	95-07935-G3		95-07944-G3	Methods Blank (one per batch)
	95-07935-G4		95-07944-G4	Matrix Spike (one per batch)
	95-07935-G5		95-07944-G5	Blank Spike (one per batch)
Fusion Dissolution KOH			95-07944-H1	KOH Fusion Sample (ICP, Radchem****)
			95-07944-H2	KOH Fusion Duplicate
			95-07944-H3	Methods Blank (one per batch)
			95-07944-H5	Post Digestion Blank Spike (ICP only)
			95-07944-H6	Post Digestion Spike (ICP Only)
Carbon	95-07935-J1		95-07944-J1	Carbon Analysis Sample (TIC/TOC/TC)
	95-07935-J2		95-07944-J2	Carbon Analysis Duplicate
	95-07935-J3		95-07944-J3	Methods Blank (one per batch)
	95-07935-J4		95-07944-J4	Matrix Spike (one per batch)
Fusion Dissolution Na2O2			95-07944-N1	Na2O2 Fusion Sample (ICP)
			95-07944-N2	Na2O2 Fusion Duplicate (one per batch)
			95-07944-N3	Methods Blank (one per batch)
			95-07944-N5	Post Digestion Blank Spike (ICP only)
			95-07944-N6	Post Digestion Spike

* Acid Digestion Radchem: Total Alpha, Total Beta, GEA
** Water Leach: IC on Drainable Liquid and Quarter Segment
*** Direct Samples: DSC on Drainable Liquid, DSC and Density on Quarter Segment
**** Fusion Radchem: Total Alpha, Total Beta, Sr-90, PU-239/240, GEA, U)

Table I-4: Tank BY-108, Core 99, Segment 4

Analysis	Quarter Segment 4A Unhomogenized	Quarter Segment 4B Unhomogenized	Quarter Segment 4C Unhomogenized	Quarter Segment 4D Unhomogenized	Quarter Segment 4A	Quarter Segment 4B	Quarter Segment 4C	Quarter Segment 4D	
TGA	95-07937	95-07938	95-07939	95-07940					Run in Duplicate
Water Leach					95-07945-C1	95-07946-C1	95-07947-C1	95-07948-C1	Water Leach Sample (IC)
					95-07945-C2	95-07946-C2	95-07947-C2	95-07948-C2	Water Leach Duplicate
					95-07945-C3	95-07946-C3	95-07947-C3	95-07948-C3	Methods Blank (one per batch)
					95-07945-C4	95-07946-C4	95-07947-C4	95-07948-C4	Spike (IC Only) (one per batch)
					95-07945-C5	95-07946-C5	95-07947-C5	95-07948-C5	Blank Spike (IC Only) (one per batch)
Direct					95-07945-D1	95-07946-D1	95-07947-D1	95-07948-D1	DSC & Density Sample
					95-07945-D2	95-07946-D2	95-07947-D2	95-07948-D2	DSC & Density Duplicate
					95-07945-D3	95-07946-D3	95-07947-D3	95-07948-D3	Methods Blank (one per batch)
Total Cyanide					95-07945-G1	95-07946-G1	95-07947-G1	95-07948-G1	Total CN Sample
					95-07945-G2	95-07946-G2	95-07947-G2	95-07948-G2	Total CN Duplicate
					95-07945-G3	95-07946-G3	95-07947-G3	95-07948-G3	Methods Blank (one per batch)
					95-07945-G4	95-07946-G4	95-07947-G4	95-07948-G4	Matrix Spike (one per batch)
					95-07945-G5	95-07946-G5	95-07947-G5	95-07948-G5	Blank Spike (one per batch)
Homogenization Test Fusion Dissolution KOH						95-07946-H1T			Homogenization Test Fusion Sample - Top *
						95-07946-H2T			Homogenization Test Fusion Duplicate - Top
						95-07946-HCB			Homogenization Test Fusion Blank
						95-07946-H1B			Homogenization Test Fusion Sample - Bottom
						95-07946-H2B			Homogenization Test Fusion Duplicate - Bottom
Fusion Dissolution KOH					95-07945-H1	95-07946-H1	95-07947-H1	95-07948-H1	KOH Fusion Sample (ICP, Radchem**)
					95-07945-H2	95-07946-H2	95-07947-H2	95-07948-H2	KOH Fusion Duplicate
					95-07945-H3	95-07946-H3	95-07947-H3	95-07948-H3	Methods Blank (one per batch)
					95-07945-H5	95-07946-H5	95-07947-H5	95-07948-H5	Post Digestion Blank Spike (ICP only)
					95-07945-H6	95-07946-H6	95-07947-H6	95-07948-H6	Post Digestion Matrix Spike (ICP Only)
Carbon					95-07945-J1	95-07946-J1	95-07947-J1	95-07948-J1	Carbon Analysis Sample (TIC/TOC/TC)
					95-07945-J2	95-07946-J2	95-07947-J2	95-07948-J2	Carbon Analysis Duplicate
					95-07945-J3	95-07946-J3	95-07947-J3	95-07948-J3	Methods Blank (one per batch)
					95-07945-J4	95-07946-J4	95-07947-J4	95-07948-J4	Matrix Spike (one per batch)
Fusion Dissolution Na2O2					95-07945-N1	95-07946-N1	95-07947-N1	95-07948-N1	Na2O2 Fusion Sample (ICP)
					95-07945-N2	95-07946-N2	95-07947-N2	95-07948-N2	Na2O2 Fusion Duplicate (one per batch)
					95-07945-N3	95-07946-N3	95-07947-N3	95-07948-N3	Methods Blank (one per batch)
					95-07945-N5	95-07946-N5	95-07947-N5	95-07948-N5	Post Digestion Blank Spike (ICP only)
					95-07945-N6	95-07946-N6	95-07947-N6	95-07948-N6	Post Digestion Spike

* Homogenization Test: ICP & GEA
** Fusion Radchem: Total Alpha, Total Beta, Sr-90, PU-239/240, GEA, U)

SECTION 1

PHYSICAL DATA

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Samples from Tank 241-BY-108 Core 99 were transferred from Westinghouse Hanford Company 222-S Laboratory to the Process Chemistry Group at the Pacific Northwest Laboratory. The segment identification, description of the sample, ACL sample number, WHC vial numbers, sample weight, and shipment numbers and dates are reported in Table 1-1.

Table 1-1: Tank BY-108, Core 99 Sample Receipt Information

Segment ID	Description	ACL Sample Number	WHC Vial Number	Approx. Sample Weight	Shipment Number	Shipment Date
1	Unhomogenized TGA	95-07931	7313	3.3 g	95-S-089	8/15/95
1	Subsampled Solids	95-07941	7504	53.1 g	95-S-089	8/15/95
2	Drainable Liquid	95-07932	7397	230 mL	95-S-093	8/18/95
2A	Subsampled Solids	95-07942	7506	7.1 g	95-S-093	8/18/95
2A	Unhomogenized TGA	95-07933	7315	1.9 g	95-S-093	8/18/95
2D	Subsampled Solids	95-07943	7505	24.0 g	95-S-093	8/18/95
2D	Unhomogenized TGA	95-07934	7314	4.0 g	95-S-093	8/18/95
3	Drainable Liquid	95-07935	7398	25 mL	95-S-094	8/21/95
3A	Subsampled Solids	95-07944	7379	18.9 g	95-S-094	8/21/95
3A	Unhomogenized TGA	95-07936	7316	3.5 g	95-S-094	8/21/95
4A	Subsampled Solids	95-07945	7716	30.3 g	95-S-095	8/25/95
4A	Unhomogenized TGA	95-07937	7322	4.0 g	95-S-095	8/25/95
4B	Subsampled Solids	95-07946	7717	33.4 g	95-S-095	8/25/95
4B	Unhomogenized TGA	95-07938	7319	4.3 g	95-S-095	8/25/95
4C	Subsampled Solids	95-07947	7718	34.0 g	95-S-095	8/25/95
4C	Unhomogenized TGA	95-07939	7318	4.6 g	95-S-095	8/25/95
4D	Subsampled Solids	95-07948	7719	32.4 g	95-S-095	8/25/95
4D	Unhomogenized TGA	95-07940	7317	5.3 g	95-S-095	8/25/95

The unhomogenized TGA samples were taken to Room 517 in the 325 Building where thermal analyses were performed. The remaining samples were homogenized using PNL technical procedure PNL-ALO-135. The sample mass of the subsampled solids from the quarter segments was small; therefore, these samples were homogenized by stirring the sample with a micro spatula. A magnetic stir bar and plate was used to homogenize the drainable liquid samples. The drainable liquids were subsampled to produce TGA samples. These TGA samples were transferred to Room 517 for thermal analysis.

The effectiveness of the homogenization process was verified on two samples (Segments 1 and 4B). GEA and ICP analyses were performed in duplicate on samples from the top and bottom portions of the homogenized samples. The major components and their concentrations for these homogenization subsamples are reported in Table 1-2.

Table 1-2: Tank BY-108, Core 99 Homogenization Check Results

Analyte	Segment	Top Sample	Duplicate	Average	RPD (%)	Bottom Sample	Duplicate	Average	RPD (%)
Cs-137, µCi/g	1	20.54	18.08	19.31	13	24.01	20.35	22.18	17
Al, µg/g	1	138516	133408	135962	4	143659	140111	141885	3
Ca, µg/g	1	1156	1450	1303	23	1330	1520	1425	13
Fe, µg/g	1	1494	972	1233	42	1805	1179	1492	42
Na, µg/g	1	98650	97695	98173	1	98980	93886	96433	5
P, µg/g	1	24562	30291	27427	21	29282	28382	28832	3
Cs-137, µCi/g	4B	115.4	91.83	103.62	23	111.7	101.8	106.75	9
Al, µg/g	4B	13405	10948	12177	20	11113	11594	11354	4
Bi, µg/g	4B	3120	2458	2789	24	2697	2753	2725	2
Ca, µg/g	4B	12280	9029	10655	31	10657	11329	10993	6
Fe, µg/g	4B	29935	22647	26291	28	25898	27279	26589	5
Na, µg/g	4B	102423	122477	112450	18	97416	104472	100944	7
P, µg/g	4B	20774	29818	25296	36	20644	23052	21848	11
Sr, µg/g	4B	27740	19740	23740	34	25301	25108	25205	1
U, µg/g	4B	61637	47328	54483	26	52421	53510	52966	2

Sample Preparation: The work scope for Tank BY-108 is comprised of segment, quarter segment and drainable liquid samples. These samples were transferred from the HLRF to the SAL. Due to the high level of radioactivity associated with the samples from Tank BY-108, all of the analytical preparations were completed in the hot cell.

Table 1-3 lists the procedures that were used to prepare Tank BY-108 samples for the requested suite of analyses. Also included in the listing is the carbon procedure that was used to conduct in-cell analytical carbon determinations.

Table 1-3: Tank BY-108, Core 99 SAL Procedure List

PNL Procedure Number	Procedure Title
PNL-ALO-103, Rev. 1	Water Leach of Sludges, Soils and Other Solid Samples
PNL-ALO-114, Rev. 1	Solubilization of Metals From Solids Using a Na_2O_2-NaOH Fusion
PNL-ALO-115, Rev. 1	Solubilization of Metals From Solids Using a $KOH-KNO_3$ Fusion
PNL-ALO-128, Rev. 0	HNO_3-HCl Acid Extraction of Liquids for Metals Analysis Using a Dry-Block Heater
PNL-ALO-129, Rev. 0	HNO_3-HCL Acid Extraction of Solids Using a Dry-Block Heater
PNL-ALO-285, Rev. 0	Total Cyanide by Remote Microdistillation and Argentometric Titration
PNL-ALO-381, Rev. 1	Direct Determination of TC, TOC, and TIC in Radioactive Sludges and Liquids by Hot Persulfate Method

Segment 1 and Quarter Segment 4B homogenization check samples were prepared for ICP and GEA by Ni/$KOH-KNO_3$ fusion. Portions of the same segment/quarter segment were acid digested for solubility observations. Segment 1 acid dissolution resulted in a clear solution with a few milligrams of gray solids on the container bottom. Segment 4B acid dissolution resulted in a clear solution with a layer of gray or reddish particulates at the top of the aqueous layer. When disturbed, the layer moved downward to the container bottom subsequently turning the entire solution very cloudy. The particulates remained suspended for hours before reforming in a layer at the top of the container.

Table 1-4 describes subjective visual data for each of the 10 segment/quarter segments samples received from the HLRF.

Table 1-4: Tank BY-108, Core 99 Visual Observations

Segment/Quarter Segment Identification	Observation
Segment 1 Solids	Beige colored, concrete textured sludge with dark colored flecks throughout.
Segment 2 Drainable Liquid	Approximately 200 mL of clear liquid with a thin layer of light beige colored solids on the bottom of the container.
Quarter Segment 2A Solids	Appearance like that of ground ice. Slides across container bottom in the same manner as wet ice.
Quarter Segment 2D Solids	Sandy beige colored sludge with a small 1/8" layer of clear liquid standing on top.
Segment 3 Drainable Liquid	Approximately 50 mL of clear liquid with a thin layer of reddish colored solids on the bottom of the container.
Quarter Segment 3A Solids	Moist, concrete textured, reddish brown sludge
Quarter Segment 4A Solids	Dark, sticky, thick, red sludge
Quarter Segment 4B Solids	Dark, sticky, thick, red sludge
Quarter Segment 4C Solids	Dark, sticky, thick, red sludge
Quarter Segment 4D Solids	Dark, sticky, thick, red sludge

Sample preparations/distributions of two drainable liquids and 8 solids samples involved:

- Water leaching of liquid and solid samples for IC and ICP.
- Ni/KOH-KNO_3 fusions of solid samples for ICP, GEA, Total Alpha, Total Beta, AEA for Pu,Np,Am, Uranium, and Sr/Y-90.
- Zr/Na_2O_2-NaOH fusions of solid samples for ICP.
- Acid digestions of liquid samples for ICP, Total Alpha, Total Beta and GEA.

- Microdistillation of liquid and solid samples for Cyanide.

Predigestion spikes were performed for ICP metals (drainable liquid samples only), IC anions, cyanide, and TOC (hot persulfate only). Post digestion spiking, where required by the Test Instruction, was done by the functional group performing the analysis.

During sample preparation, the SAL may have made deliberate minor deviations to sample preparatory procedures for one or more of the following reasons:

- Insufficient sample was available to conduct the analyses per procedure while maintaining the level of quality control requested.
- Sample weights and/or final volumes were reduced to facilitate waste minimization.
- Sample weights and/or final volumes were altered to increase the concentration of certain analytes of interest. This was done to meet the procedural concentration ranges needed to perform the analyses.

Sample sizes and final volumes for all sample preparations are documented on the Sample Preparation Data Sheets included in Appendix A. Table 1-5 lists the sample preparatory procedure deviations performed during the processing of Tank BY-108.

Table 1-5: Tank BY-108, Core 99 Sample Preparation Procedural Deviations

ALO Number	Prep Method	Sample Size Deviation	Sample Volume Deviation	Reagent Deviation	Observed Effect
95-07932-A	Acid	Yes	No	No	None
95-07935-A	Acid	Yes	No	No	None
95-07932-C	Water	Yes	Yes	No	None
95-07935-C	Water	Yes	Yes	No	None
95-07941-C	Water	No	Yes	No	None
95-07942-C	Water	Yes	Yes	No	None
95-07944-C	Water	Yes	Yes	No	None
95-07945-C	Water	Yes	Yes	No	None
95-07946-C	Water	Yes	Yes	No	None
95-07947-C	Water	Yes	Yes	No	None
95-07948-C	Water	Yes	Yes	No	None

Thermal Analysis

Scanning Thermogravimetric Analysis (TGA) and Differential Scanning Calorimetry (DSC) were performed on the unhomogenized solids and drainable liquids from Core 99 Segments 1 through 4. These two thermal analysis techniques are useful in determining the thermal stability and reactivity of the material. DSC measures heat released or absorbed while the temperature of the sample is increased at a constant rate. Data generated by the DSC analysis is often used to measure thermal decomposition temperatures, heats of reaction, reaction temperatures, melting points and solid-solid transition temperatures. TGA measures the mass of a sample while the temperature of the sample is increased at a constant rate. The TGA data is used to measure thermal decomposition temperatures, water content and reaction temperatures. Both methods can be modified to measure isothermal changes in the material and provide complimentary information.

The calibration of the DSC and TGA instruments were checked before running these samples. An indium standard was run on the DSC to check the temperature and enthalpy calibrations. The balance calibration of the TGA was checked with a 100 mg standard weight, and the temperature calibration was checked with a lead standard. The temperature and enthalpy calibration checks were all within 1°C and 0.2 J/g of their reported values, and the balance calibration was within 0.01 mg.

The results from the DSC and TGA analyses are reported in Tables 1-6 and 1-7, respectively. The temperature range of the scans was from ambient to 550°C, with a scan rate of 10°C per minute. These analyses were all performed in platinum pans under nitrogen cover gas.

The major transition in all of these samples was an endotherm due to water loss from the sample. This was also the major mass loss in all of the samples. The onset temperature of this transition could not be measured because the transition began at the initial temperature of the run (ambient temperature). The water loss endotherm ends at approximately 200°C. This endotherm is a complicated system of several unresolvable transitions with each sample containing slightly different proportions of these transitions.

An exotherm was identified in all quarter segment samples from Segment 4 and the drainable liquids. This exotherm has an onset temperature of between 197 and 225°C. The enthalpy of this transition varied with each quarter segment. The temperature range for this transition was from 195 to 400°C. It appears that this exotherm is commingled with some endothermic behavior which cannot be resolved; therefore, it is difficult to determine onset temperatures for each sample. The enthalpy of this transition is also dependent upon resolution of this exotherm for the commingled endotherms and the initial water loss endotherm. In most cases the most conservative approach (the largest exothermic energy) has been taken.

In Segments 1 through 3 a second endotherm is observed. Several transitions are observed in this endotherm and the onset temperature varies depending upon the size of the first transition in comparison with the second

Table 1-6: Tank BY-108, Core 99 Differential Scanning Calorimetric (DSC) Results

Sample Number	Segment ID	Enthalpy (J/g)	Range (°C)	Onset (°C)
7313	1	725.6 311.2	8-153 193-333	219
7313-2	1	521.2 305.2	16-158 181-343	221
95-07932	2-DL	737.5 -11.6 -14.9	45-226 226-265 265-340	278
95-07932-2	2-DL	1243.6 -35.8 -10.5	20-216 216-270 272-330	279
7315	2A	201.3 108.6	48-227 227-324	250
7315-2	2A	177.3 114.1	43-231 231-322	251
7314	2D	263.0 127.3 -2.8	22-204 215-332 375-430	261 385
7314-2	2D	194.7 161.7 -2.8	24-185 187-345 380-430	211
95-07935	3-DL	1178.2 26.1	31-236 236-303	237
95-07935-2	3-DL	1279.2 26.9	32-239 239-300	238
7316	3A	303.4 81.1 -1.2	28-230 236-336 400-427	249
7316-2	3A	343.5 101.1 -0.7	12-215 233-327 430-450	259
7322	4A	385.5 33.6	22-183 243-310	259
7322-2	4A	556.7	33-255	
7319	4B	651.6 -191.1	22-193 200-411	207

Table 1-6: Tank BY-108, Core 99 Differential Scanning Calorimetric (DSC) Results (Cont.)

Sample Number	Segment ID	Enthalpy (J/g)	Range (°C)	Onset (°C)
7319-2	4B	476.0 -133.2	19-224 224-385	
7318	4C	644.5 -67.0	26-192 192-375	197
7318-2	4C	876.6 -77.8	15-196 200-361	237
7317	4D	693.4 -73.7	23-195 205-369	206
7317-2	4D	613.9 -60.2	30-193 203-350	204

transition. It appears that the onset temperature for the first transition in this endotherm is 220°C, and the onset temperature for the second transition is between 250 and 260°C.

The TGA analysis also indicated two different waste types in this core sample. All of the segments had a large mass loss associated with the water loss endotherm. This mass loss was observed between ambient temperature and 180°C. A small mass loss was observed over the remainder of the temperature range of the TGA analysis for all of the Segment 4 samples, but no significant transitions were observed at higher temperatures. In the Segment 1 through 3 solid samples, a second significant mass loss was observed. This mass loss has an onset temperature of approximately 245°C. This transition correlates with the second endothermic transition observed in the DSC analyses. These samples also continued to have a small mass loss throughout the remainder of the temperature range.

Table 1-7: Tank BY-108, Core 99 Thermogravimetric Analysis (TGA) Results

Sample	Segment	Range (°C)	Onset (°C)	Mass Loss (%)
7313	1	23-157 157-309 309-548	247	32.7 9.7 2.6
7313-2	1	28-145 145-318 318-548	249	26.1 13.2 3.5
7316	3A	22-212 212-344 344-548	310	14.1 0.9 0.2
7316	3A	40-170 170-548		35.0 1.9
7314	2D	27-198 198-328 328-545	245	17.9 2.2 0.9
7314-2	2D	23-203 203-328 328-442 442-545	242 393	15.5 3.3 0.8 0.4
7315	2A	23-136 136-256 256-545		7.8 3.6 0.8
7315-2	2A	23-130 130-215 215-545		6.6 5.2 0.1
7317	4D	23-167 167-550		40.6 5.1
7317-2	4D	22-178 178-549		44.0 3.8
7318	4C	24-173 173-549		35.6 5.3
7318-2	4C	24-172 172-549		36.2 4.5
7319	4B	22-187 187-403 403-548	309	35.3 7.9 1.3

Table 1-7: Tank BY-108, Core 99 Thermogravimetric Analysis (TGA) Results (Cont.)

Sample	Segment	Range (°C)	Onset	Mass Loss (%)
7319-2	4B	23-172 172-412 412-549	300	35.9 8.1 0.8
7322	4A	22-160 160-550		25.8 3.1
7322-2	4A	23-171 171-548		25.1 3.4
95-07935	3-DL	23-227 227-548		52.1 0.8
95-07935	3-DL	24-235 235-548		52.6 0.7
95-07932	2-DL	28-218 218-548		52.6 0.8
95-07932	2-DL	23-236 236-547		46.7 0.6

DSC/TGA SCANS

DSC

<Name>
14sept95a

<Date>
95/09/14 06:20

<Sample>
14sept95a
11.700 mg
(11.700 mg)

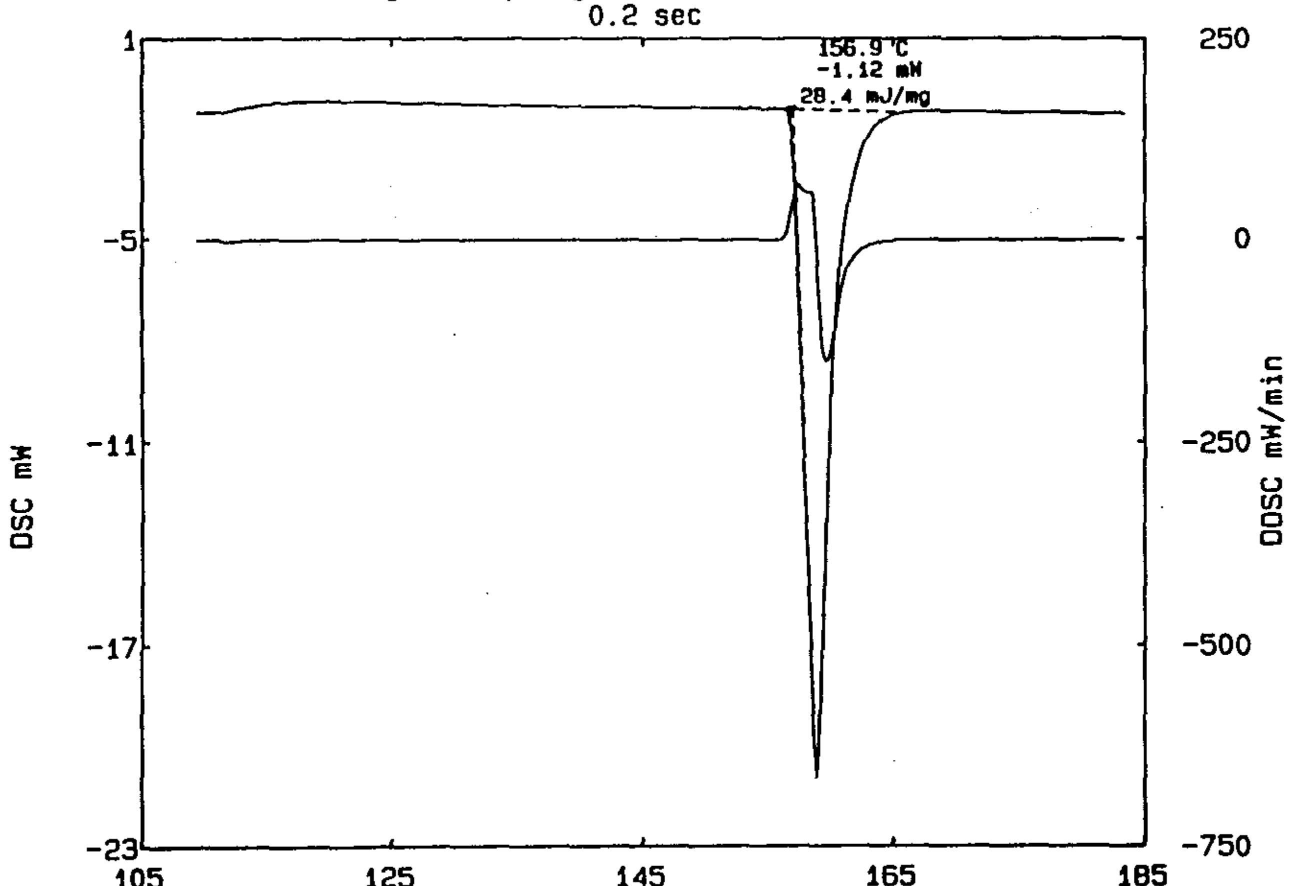
<Reference>
mt pan and lid
0.000 mg

<Comment>
indium std

<Sampling>
0.2 sec

<Temp.program (C)	[C/min]	[min]>
1 20.0- 120.0	20.00	0.00
2* 120.0- 190.0	10.00	0.00

<Gas>
nitrogen 300.0 ml/min
0.0 ml/min



Chemical Process Systems

156.6
28.59

3-30

WHC-SD-WM-DP-145, REV. 1

WHC-SD-WM-DP-145, REV. 1

TG/DTA

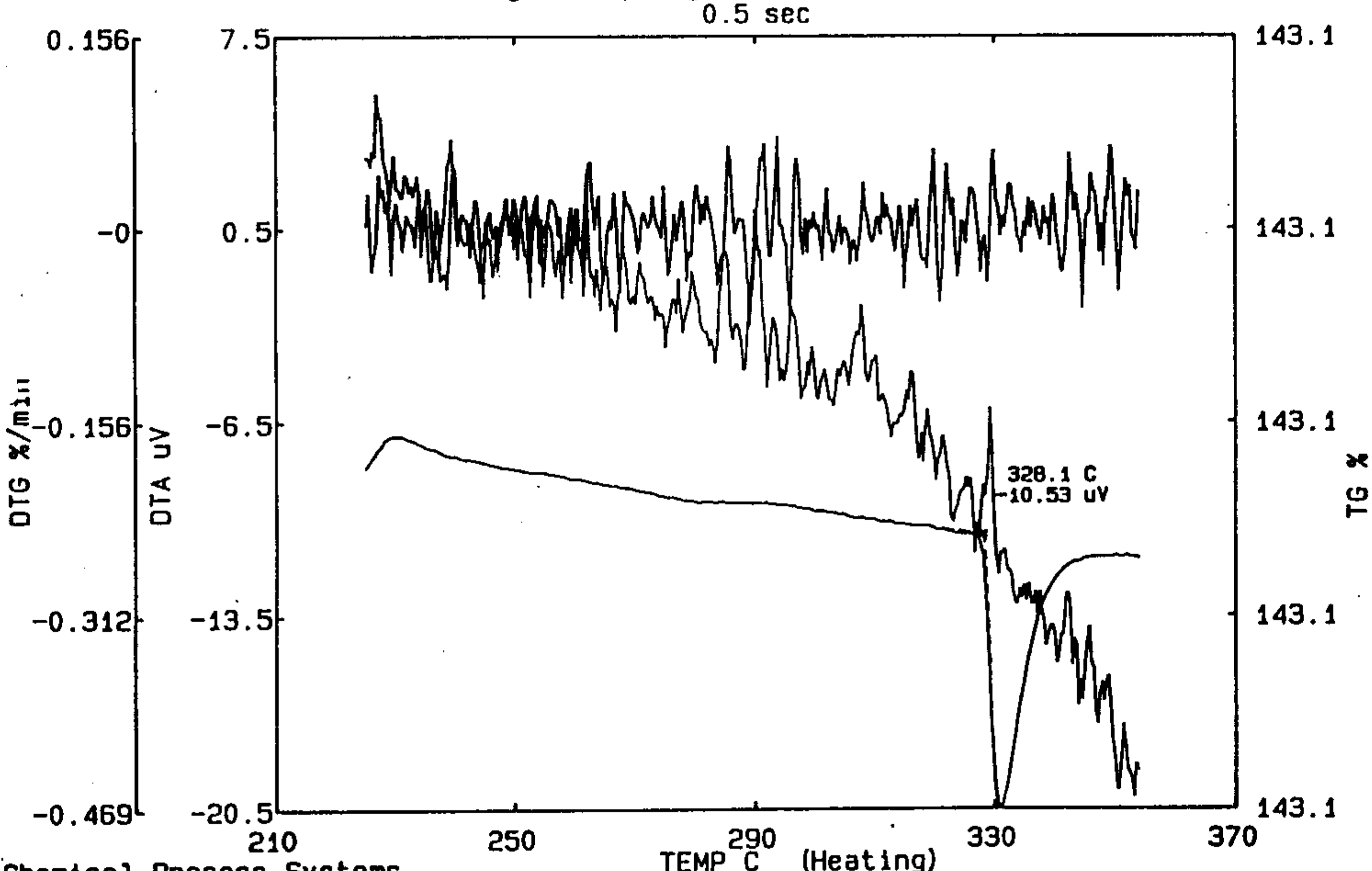
<Name>
14sept95d
<Date>
95/09/14 06:48

<Sample>
14sept95d
19.200 mg
(19.200 mg)
<Reference>
mt pan
0.000 mg

<Comment>
lead std
<Sampling>
0.5 sec

<Temp.program[C]	[C/min]	[min]>
1 20.0- 250.0	25.00	1.00
2* 250.0- 370.0	10.00	0.00

<Gas>
nitrogen 300.0 ml/min
0.0 ml/min



Chemical Process Systems

327.5
DKa

3-31

DSC

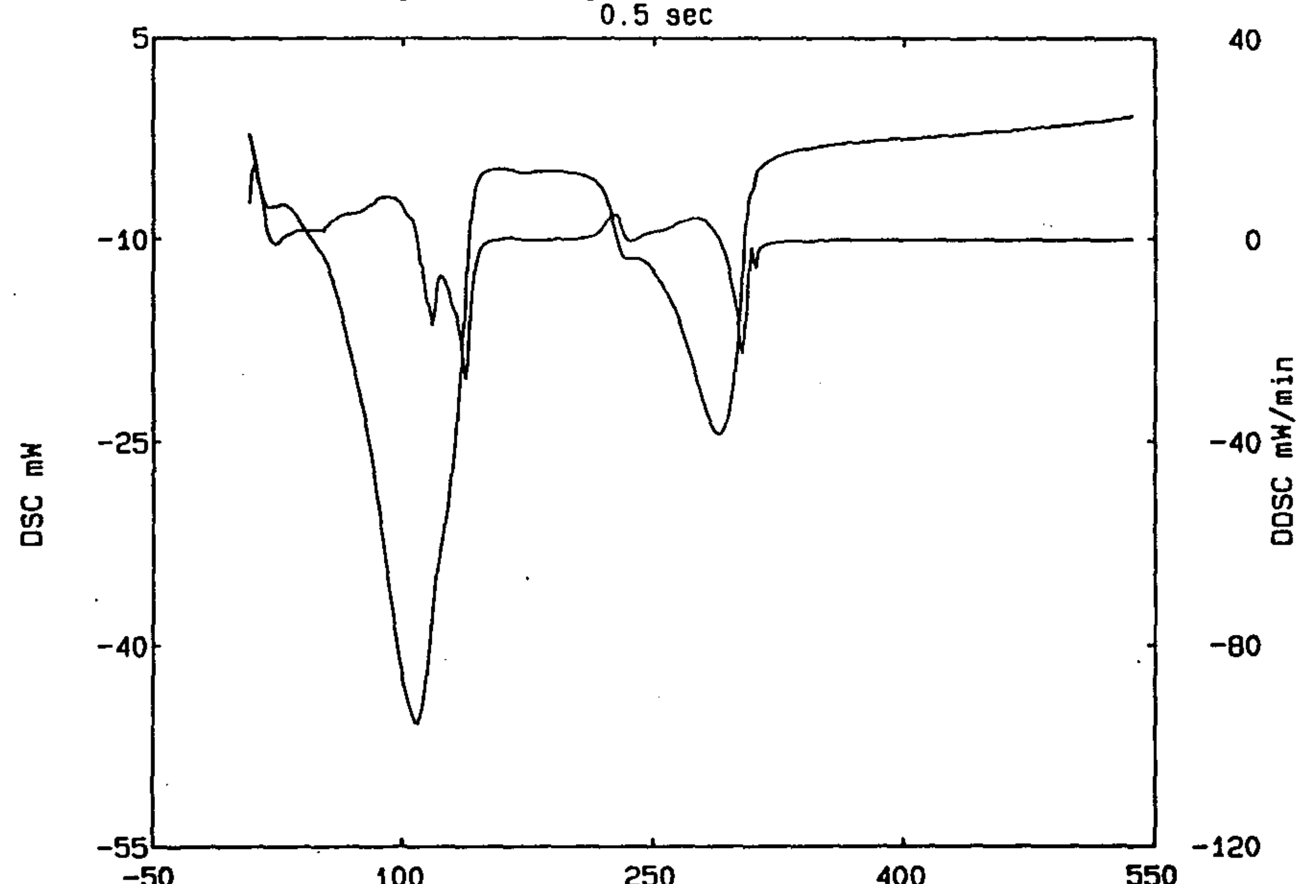
<Name>
20sept95c
<Date>
95/09/20 08:38

<Sample>
20sept95c
19.935 mg
(19.935 mg)
<Reference>
pt pan
0.000 mg

<Comment>
7313
<Sampling>
0.5 sec

<Temp.program [C]	[C/min]	[min]>
1* 20.0- 550.0	10.00	0.00

<Gas>
nitrogen 300.0 ml/min
0.0 ml/min



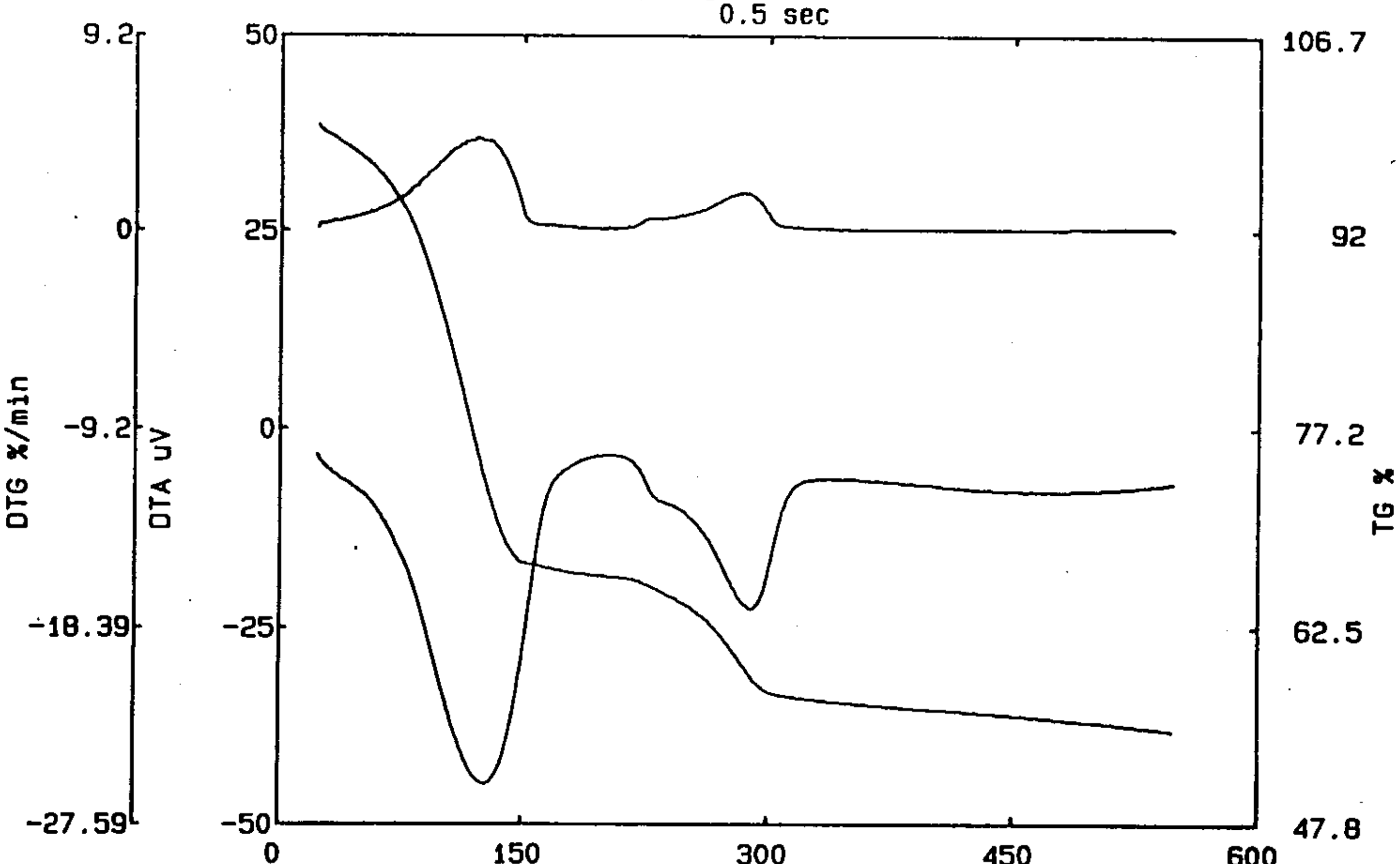
Chemical Process Systems

TG/DTA
<Name>
20sept95d
<Date>
95/09/20 08: 41

<Sample>
20sept95d
27.185 mg
(27.185 mg)
<Reference>
pt pan
0.000 mg

<Comment>
7313
<Sampling>
0.5 sec

<Temp.program [C] [C/min] [min]>
1* 20.0- 550.0 10.00 0.00
<Gas>
nitrogen 300.0 ml/min
0.0 ml/min



Chemical Process Systems

3-33

WHC-SD-WM-DP-145, REV. 1

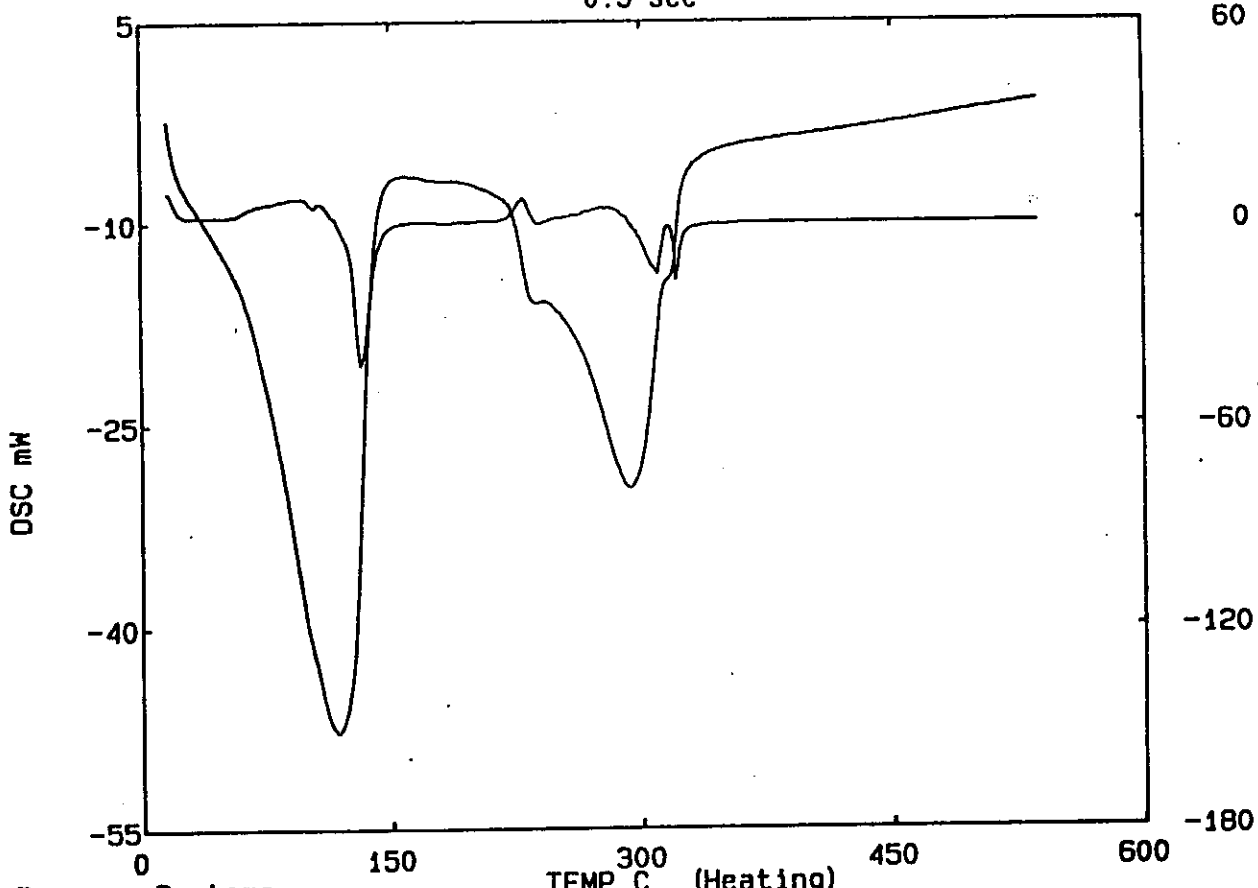
DSC

<Name>
20sept95e
<Date>
95/09/20 10:14

<Sample>
20sept95e
29.313 mg
(29.313 mg)
<Reference>
pt pan
0.000 mg

<Comment>
7313-2
<Sampling>
0.5 sec

<Temp.program [C] [C/min] [min]>
1* 20.0- 550.0 10.00 0.00
<Gas>
nitrogen 300.0 ml/min
0.0 ml/min



Chemical Process Systems

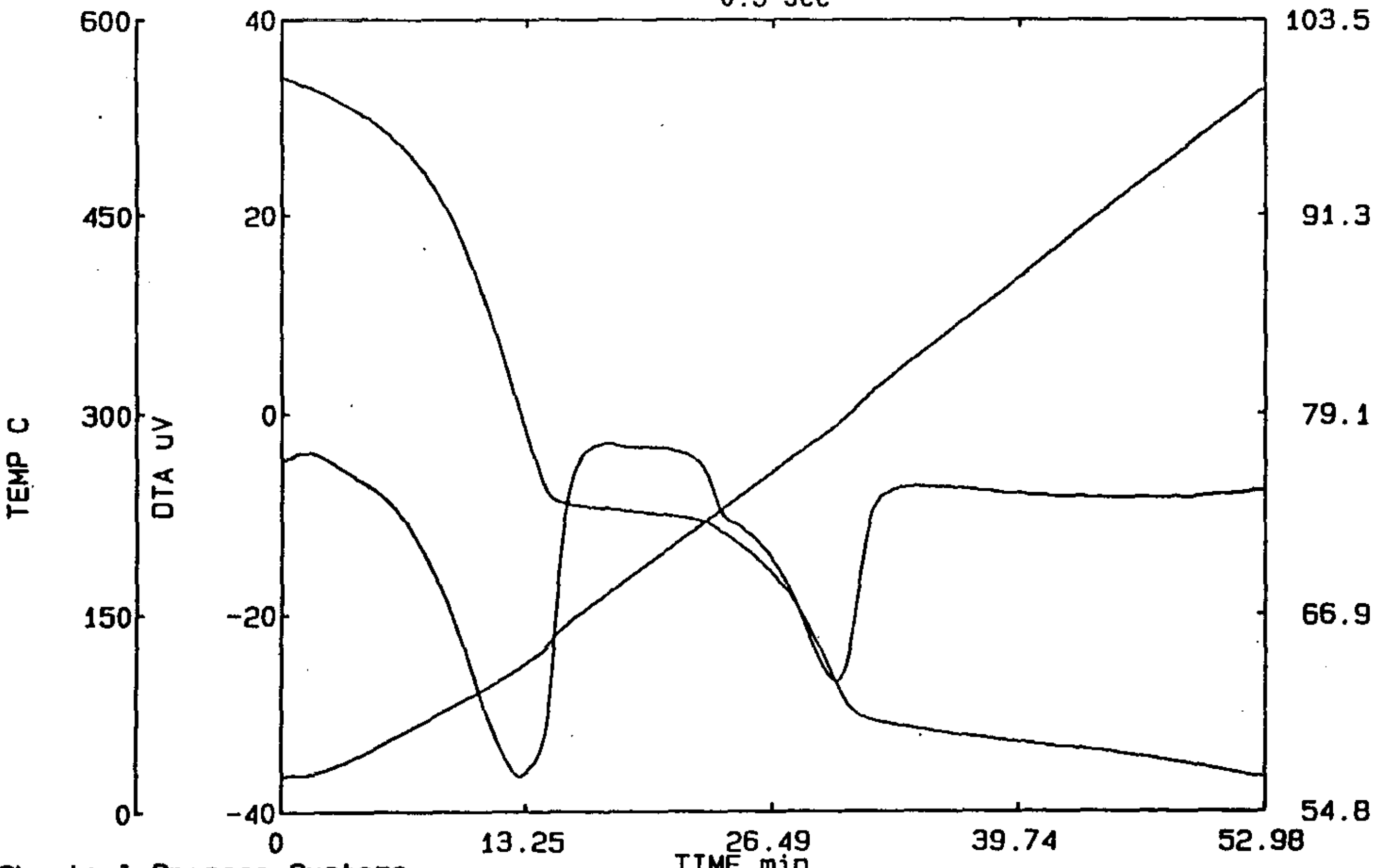
TG/DTA

<Name>
20sept95f
<Date>
95/09/20 10: 18

<Sample>
20sept95f
24.648 mg
(24.648 mg)
<Reference>
pt pan
0.000 mg

<Comment>
7313-2
<Sampling>
0.5 sec

<Temp.program [C] [C/min] [min]>
1* 20.0- 550.0 10.00 0.00
<Gas>
nitrogen 300.0 ml/min
0.0 ml/min



Chemical Process Systems

DSC

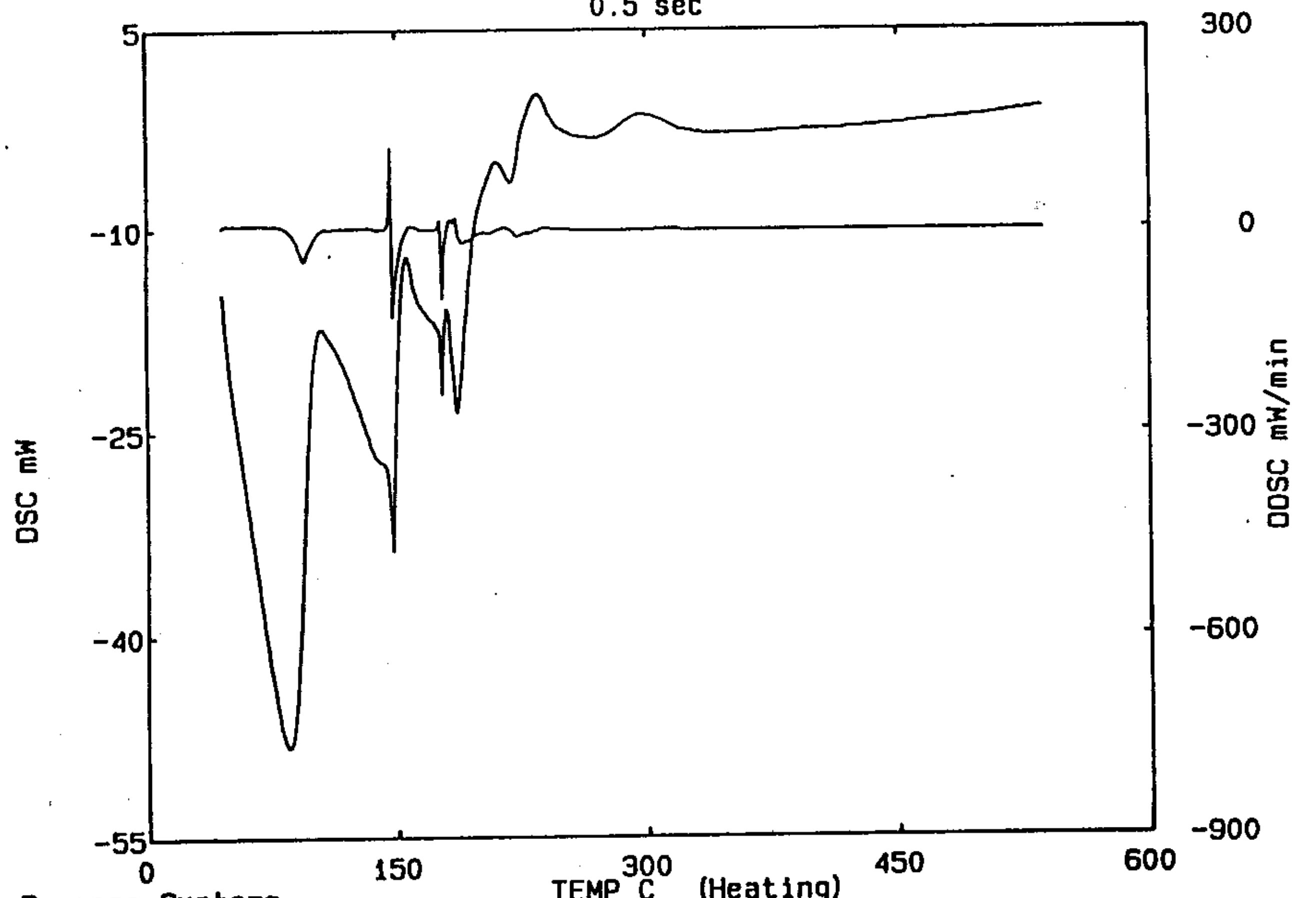
<Name>
19sept95k
<Date>
95/09/19 18:29

<Sample>
19sept95k
20.316 mg
(20.316 mg)
<Reference>
pt pan
0.000 mg

<Comment>
95-07932

<Sampling>
0.5 sec

<Temp.program [C] [C/min] [min]>
1* 20.0- 550.0 10.00 0.00
<Gas>
nitrogen 300.0 ml/min
--------------- 0.0 ml/min



Chemical Process Systems

IPM

TG/DTA

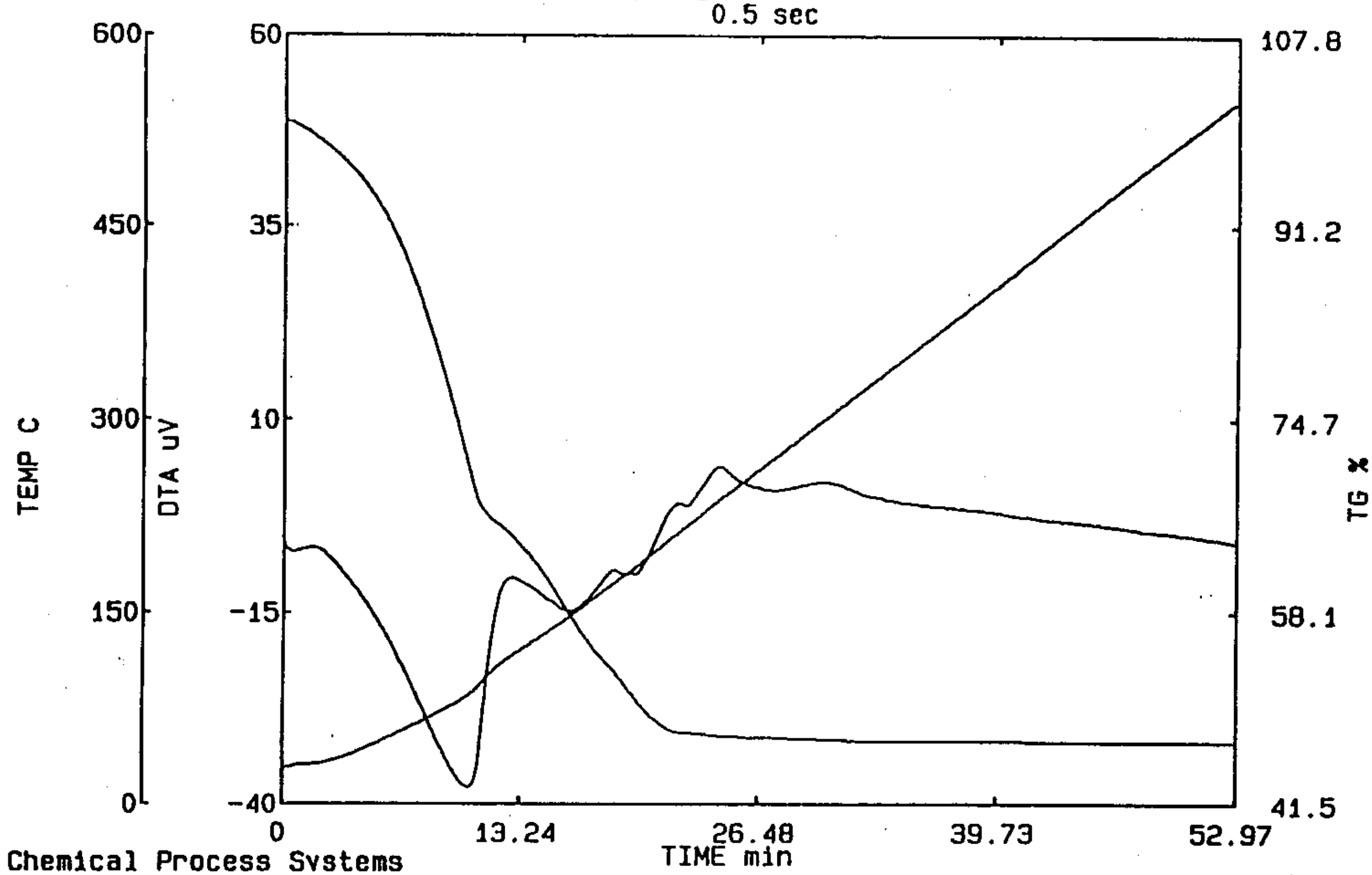
<Name>
19sept951
<Date>
95/09/19 18:32

<Sample>
19septy951
18.083 mg
(18.083 mg)
<Reference>
pt pan
0.000 mg

<Comment>
95-07932

<Sampling>
0.5 sec

<Temp.program[C] [C/min] [min]>
1* 20.0- 550.0 10.00 0.00
<Gas>
nitrogen 300.0 ml/min
---------------- 0.0 ml/min



Xa

DSC

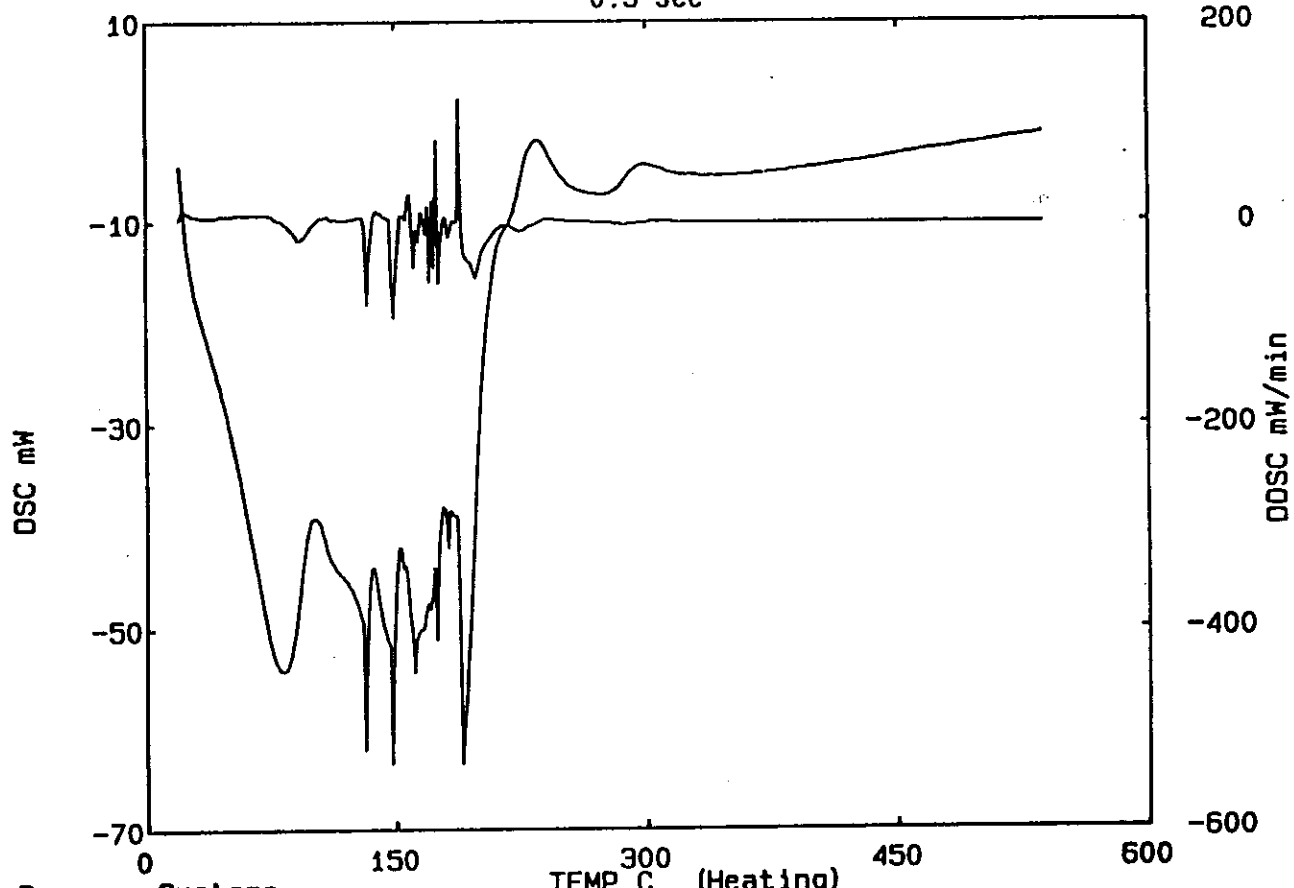
<Name>
20sept95a
<Date>
95/09/20 06:31

<Sample>
20sept95a
30.332 mg
(30.332 mg)
<Reference>
pt pan
0.000 mg

<Comment>
95-07932-2

<Sampling>
0.5 sec

<Temp.program [C] [C/min] [min]>
1* 20.0- 550.0 10.00 0.00
<Gas>
nitrogen 300.0 ml/min
---------------- 0.0 ml/min



Chemical Process Systems

IDM

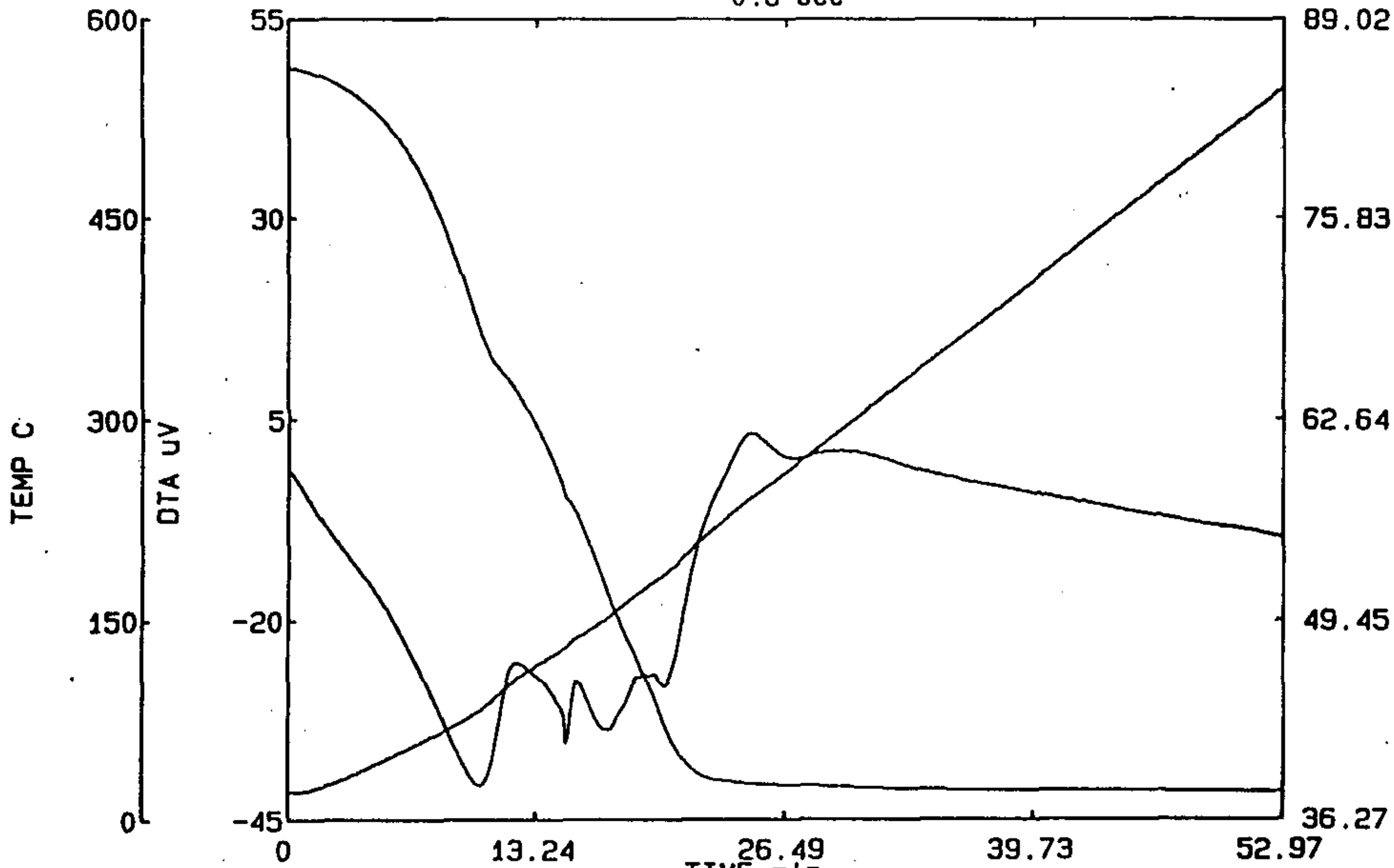
TG/DTA

<Name>
20sept95b
<Date>
95/09/20 06:32

<Sample>
20sept95b
30.332 mg
(30.332 mg)
<Reference>
pt pan
0.000 mg

<Comment>
95-07932-2
<Sampling>
0.5 sec

<Temp.program [C] [C/min] [min]>
1* 20.0- 550.0 10.00 0.00
<Gas>
nitrogen 300.0 ml/min
0.0 ml/min



Chemical Process Systems

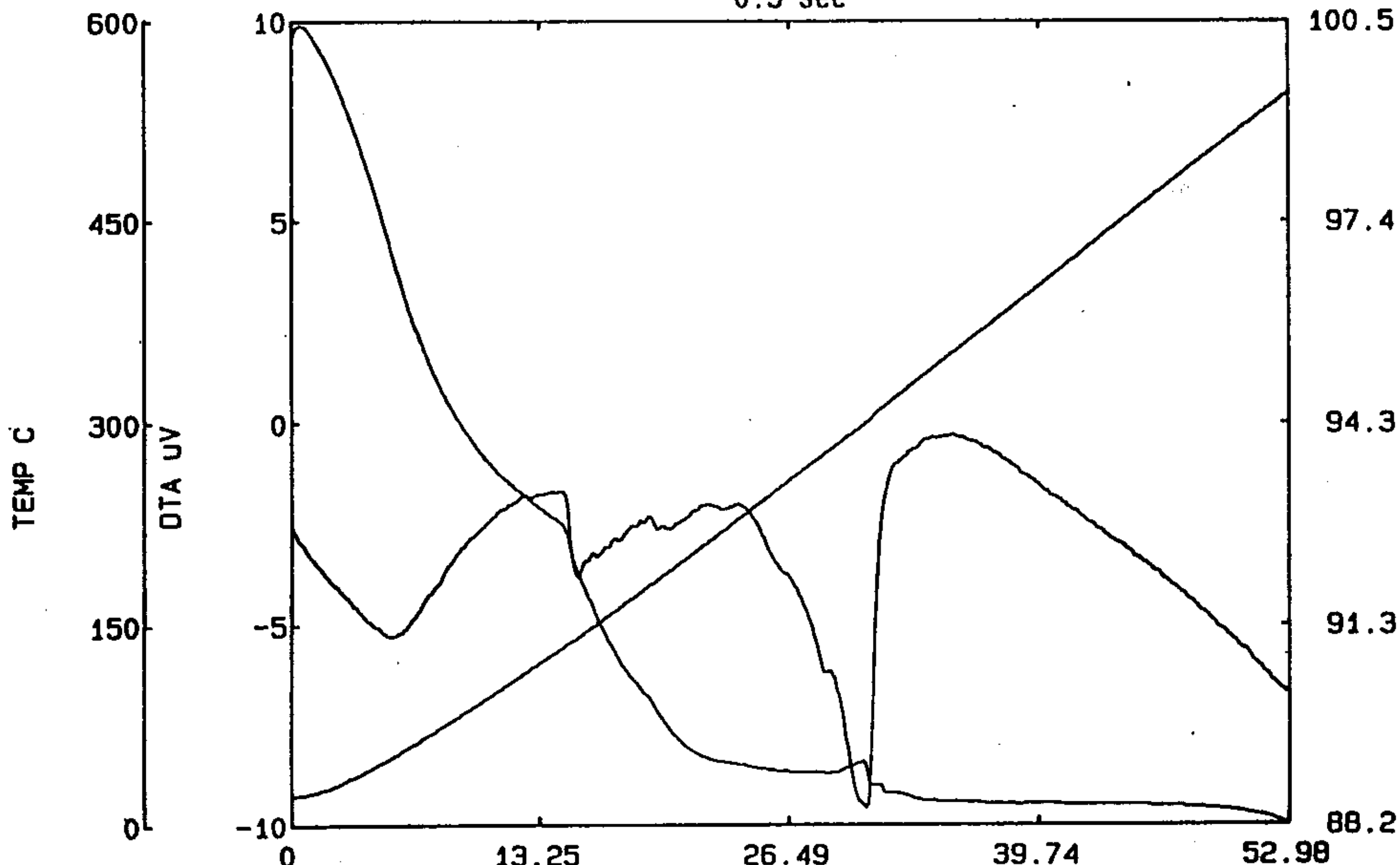
TG/DTA

<Name>
21sept95f
<Date>
95/09/21 15:07

<Sample>
21sept95f
12.984 mg
(12.984 mg)
<Reference>
pt pan
0.000 mg

<Comment>
7315
<Sampling>
0.5 sec

<Temp.program [C] [C/min] [min]>
1* 20.0- 550.0 10.00 0.00
<Gas>
nitrogen 300.0 ml/min
0.0 ml/min



Chemical Process Systems

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WHC-SD-WM-DP-145, REV. 1

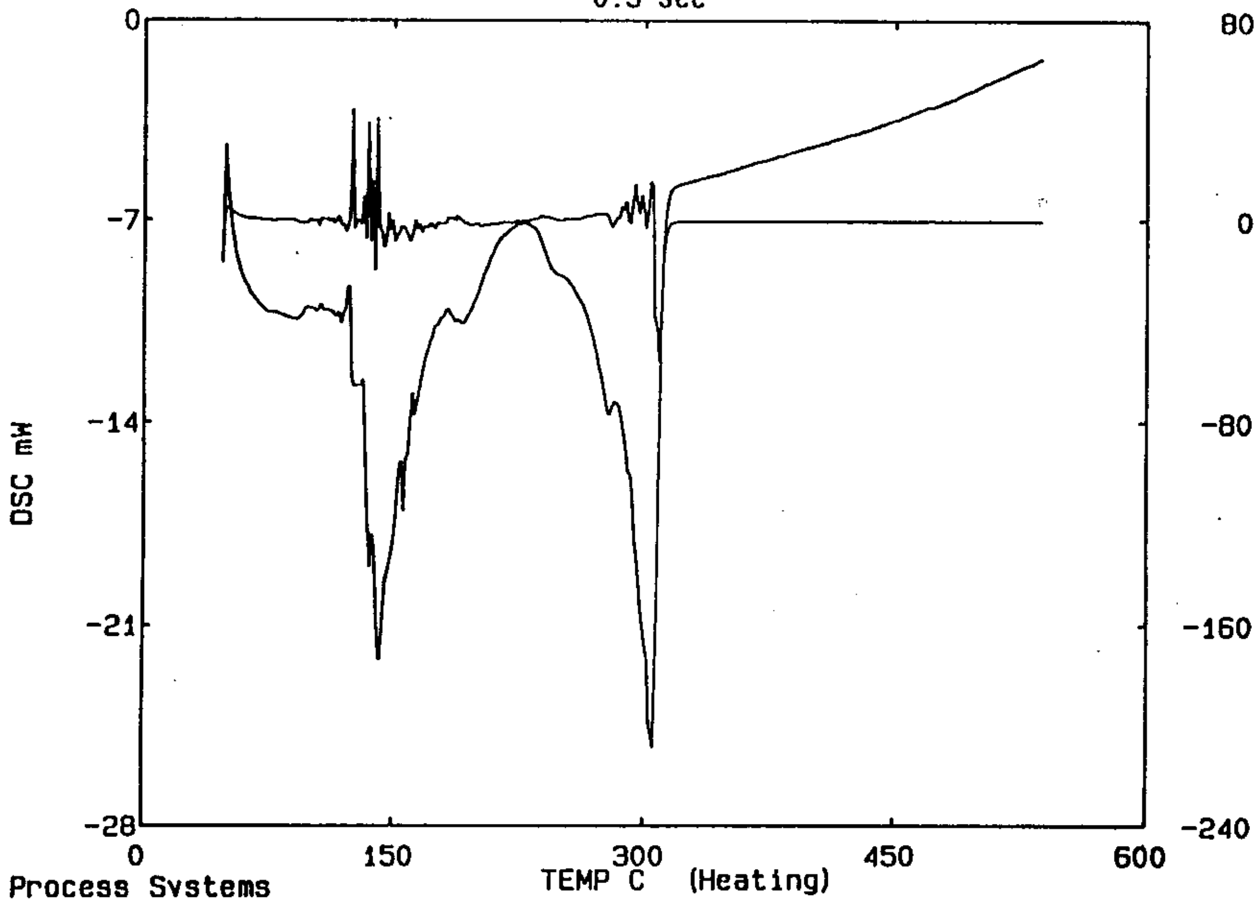
DSC

<Name>
21sept95e
<Date>
95/09/21 15:05

<Sample>
21sept95e
28.860 mg
(28.860 mg)
<Reference>
pt pan
0.000 mg

<Comment>
7315
<Sampling>
0.5 sec

<Temp.program [C] [C/min] [min]>
1* 20.0- 550.0 10.00 0.00
<Gas>
nitrogen 300.0 ml/min
0.0 ml/min



Chemical Process Systems

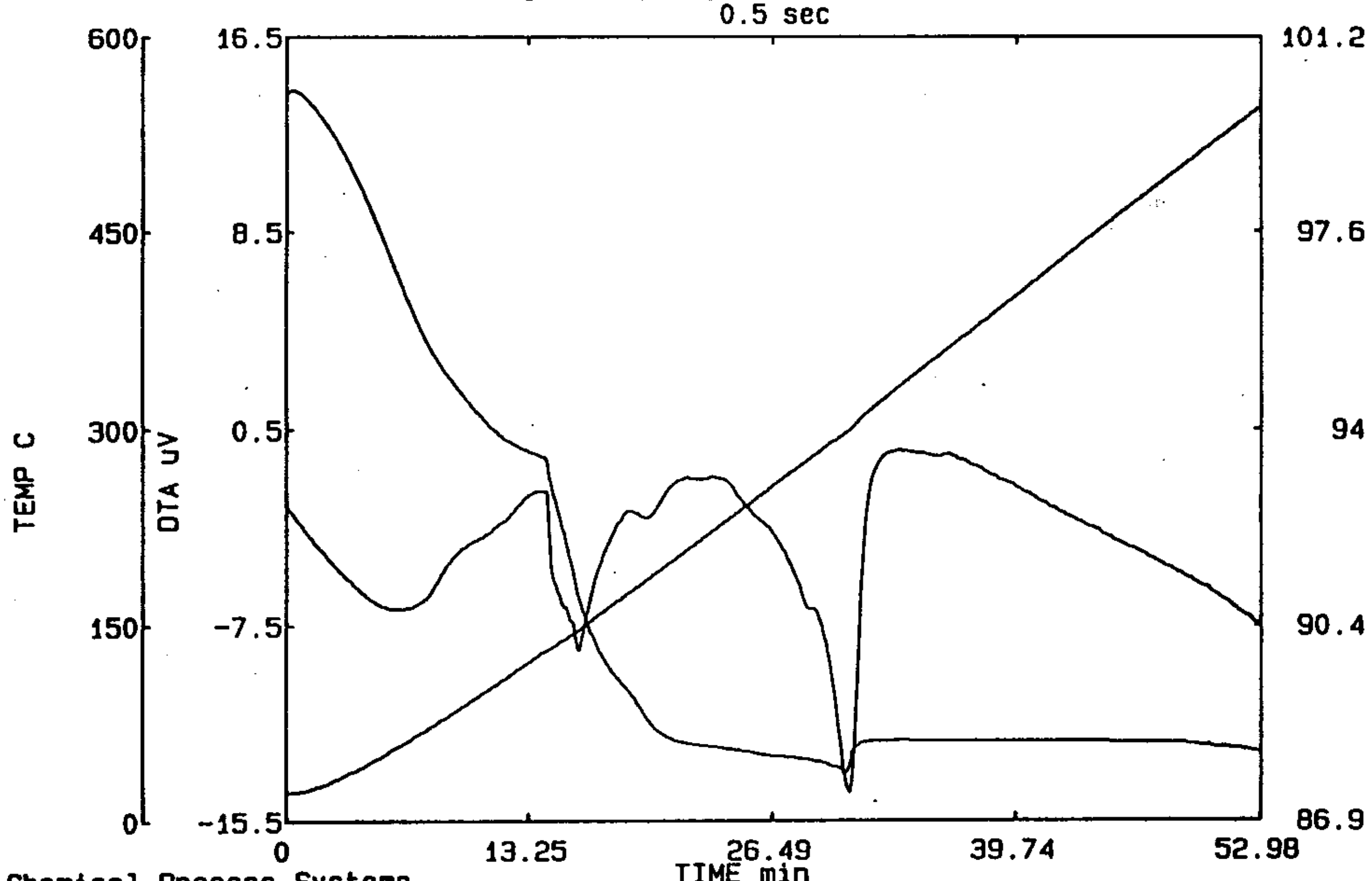
TG/DTA

<Name>
21sept95h
<Date>
95/09/21 16:57

<Sample>
21sept95h
22.391 mg
(22.391 mg)
<Reference>
pt pan
0.000 mg

<Comment>
7315-2
<Sampling>
0.5 sec

<Temp.program[C] [C/min] [min]>
1* 20.0- 550.0 10.00 0.00
<Gas>
nitrogen 300.0 ml/min
0.0 ml/min



Chemical Process Systems

WHC-SD-WM-DP-145, REV. 1

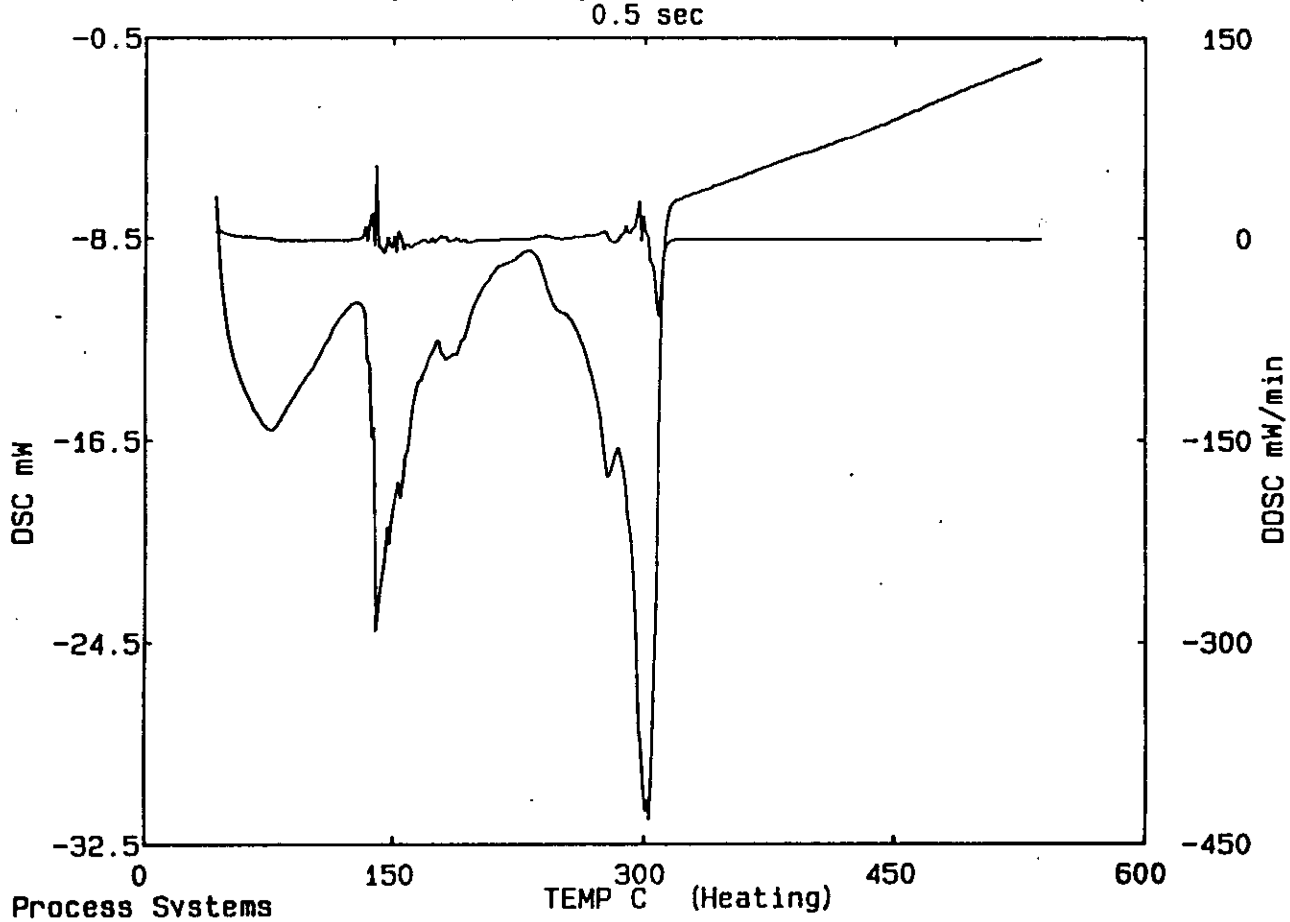
DSC

<Name>
21sept95g
<Date>
95/09/21 16:55

<Sample>
21sept95g
35.068 mg
(35.068 mg)
<Reference>
pt pan
0.000 mg

<Comment>
7315-2
<Sampling>
0.5 sec

<Temp.program [C] [C/min] [min]>
1* 20.0- 550.0 10.00 0.00
<Gas>
nitrogen 300.0 ml/min
0.0 ml/min



Chemical Process Systems

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WHC-SD-WM-DP-145, REV 1

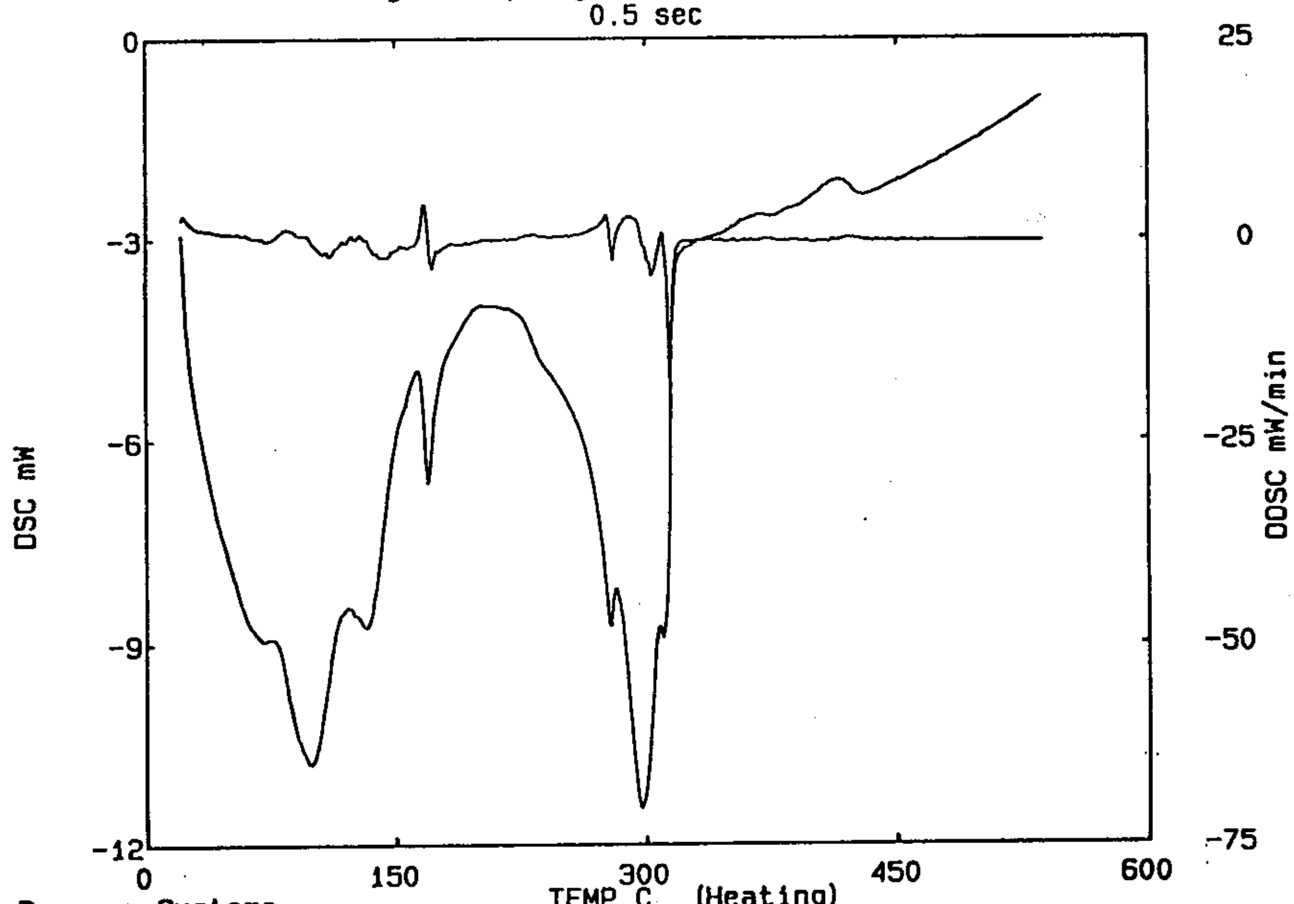
DSC

<Name>
21sept95a
<Date>
95/09/21 09:00

<Sample>
21sept95a
16.036 mg
(16.036 mg)
<Reference>
pt pan
0.000 mg

<Comment>
7314
<Sampling>
0.5 sec

<Temp.program [C] [C/min] [min]>
1* 20.0- 550.0 10.00 0.00
<Gas>
nitrogen 300.0 ml/min
0.0 ml/min



Chemical Process Systems

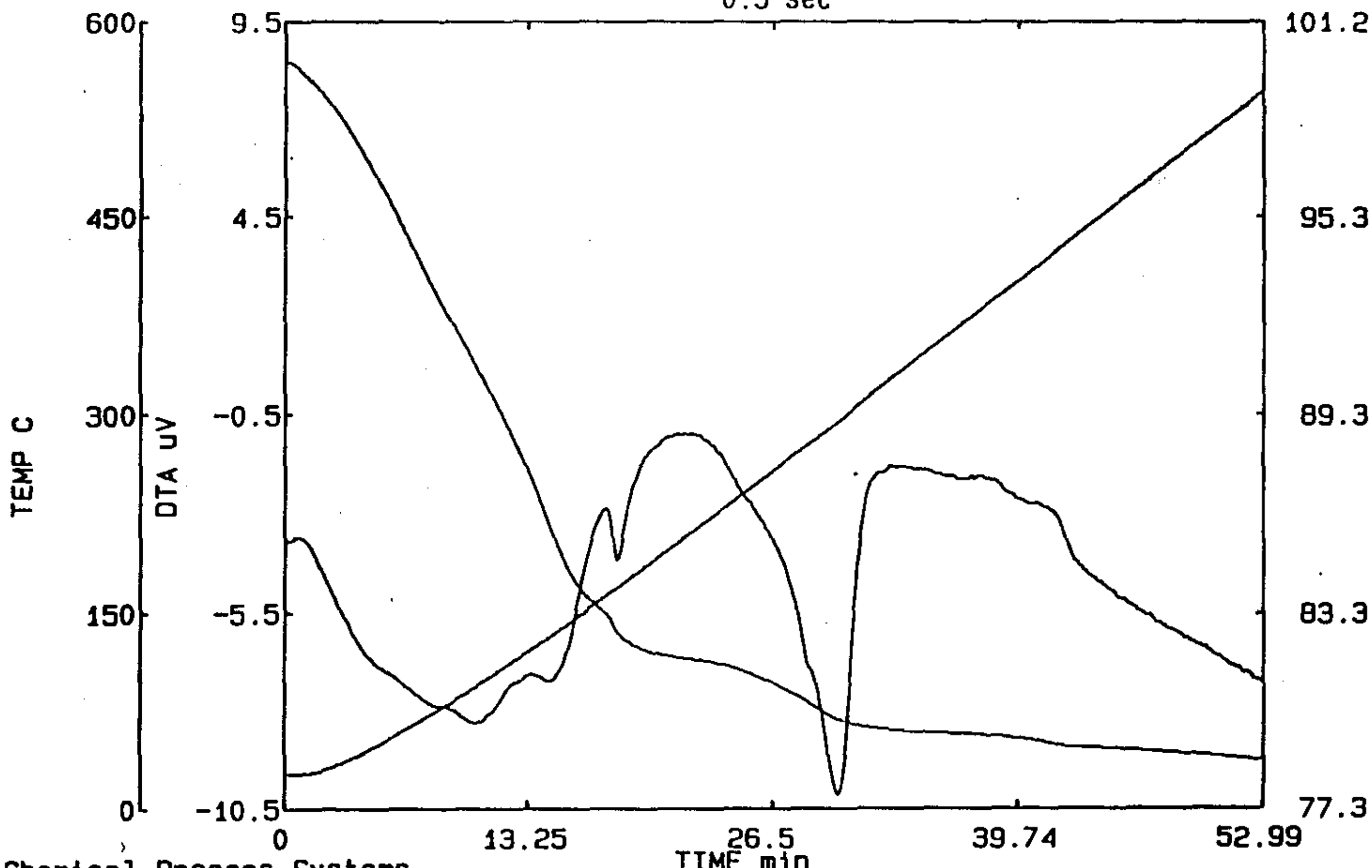
TG/DTA

<Name>
21sept95b
<Date>
95/09/21 09:04

<Sample>
21sept95b
15.063 mg
(15.063 mg)
<Reference>
pt pan
0.000 mg

<Comment>
7314
<Sampling>
0.5 sec

<Temp.program [C] [C/min] [min]>
1* 20.0- 550.0 10.00 0.00
<Gas>
nitrogen 300.0 ml/min
0.0 ml/min



Chemical Process Systems

WHC-SD-WM-DP-165, REV.

3-45

DSC

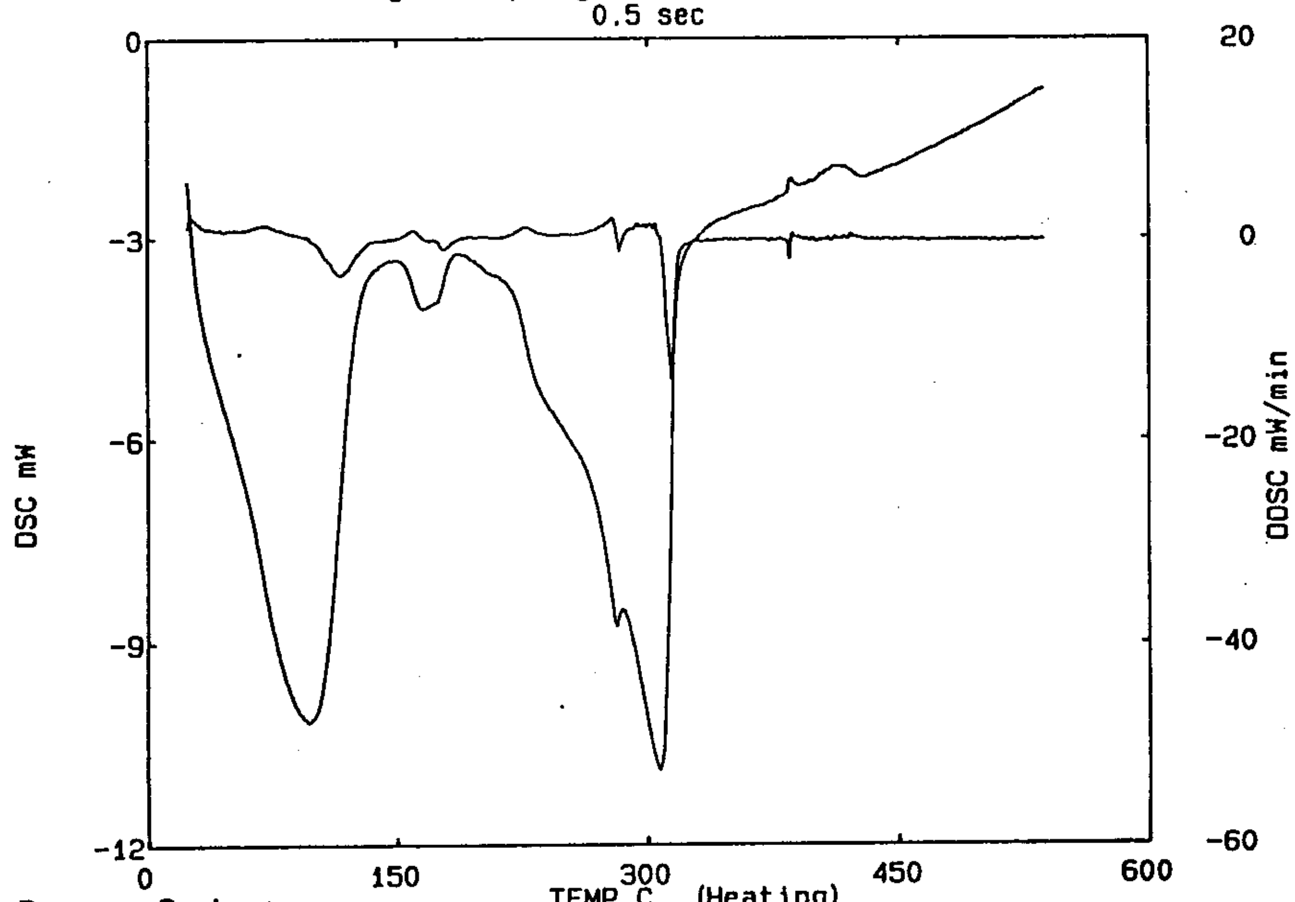
<Name>
21sept95c
<Date>
95/09/21 13:34

<Sample>
21sept95c
16.004 mg
(16.004 mg)
<Reference>
pt pan
0.000 mg

<Comment>
7314-2

<Sampling>
0.5 sec

<Temp.program [C] [C/min] [min]>
1* 20.0- 550.0 10.00 0.00
<Gas>
nitrogen 300.0 ml/min
---------------- 0.0 ml/min



Chemical Process Systems

3-46

WHC-SD-WM-DP-145, REV. 1

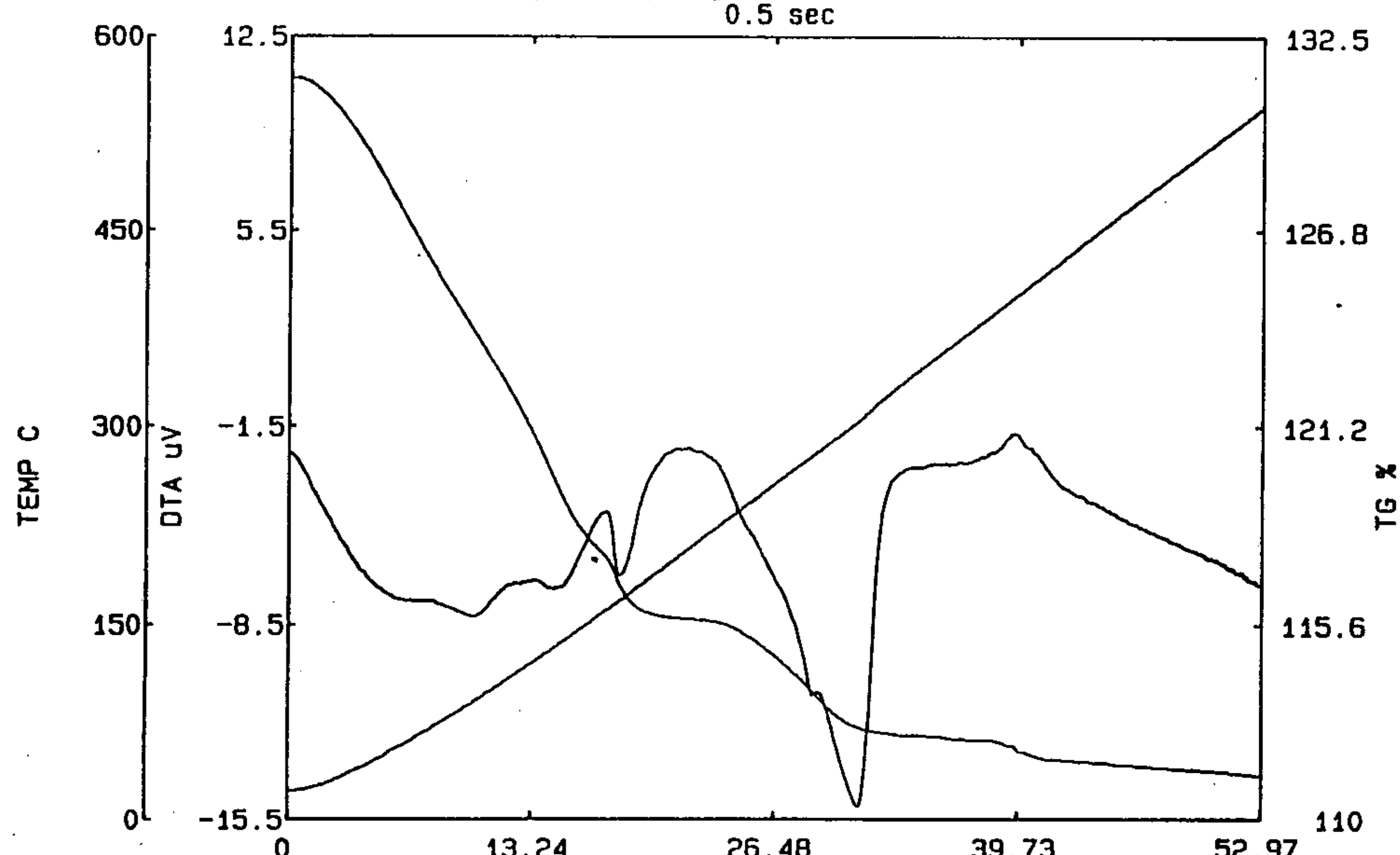
1190

TG/DTA
<Name>
21sept95d
<Date>
95/09/21 13:34

<Sample>
21sept95d
16.004 mg
(16.004 mg)
<Reference>
pt pan
0.000 mg

<Comment>
7314-2
<Sampling>
0.5 sec

<Temp.program [C] [C/min] [min]>
1* 20.0- 550.0 10.00 0.00
<Gas>
nitrogen 300.0 ml/min
0.0 ml/min



Chemical Process Systems

3- 47

WHC-SD-WM-DP-145, REV. 1

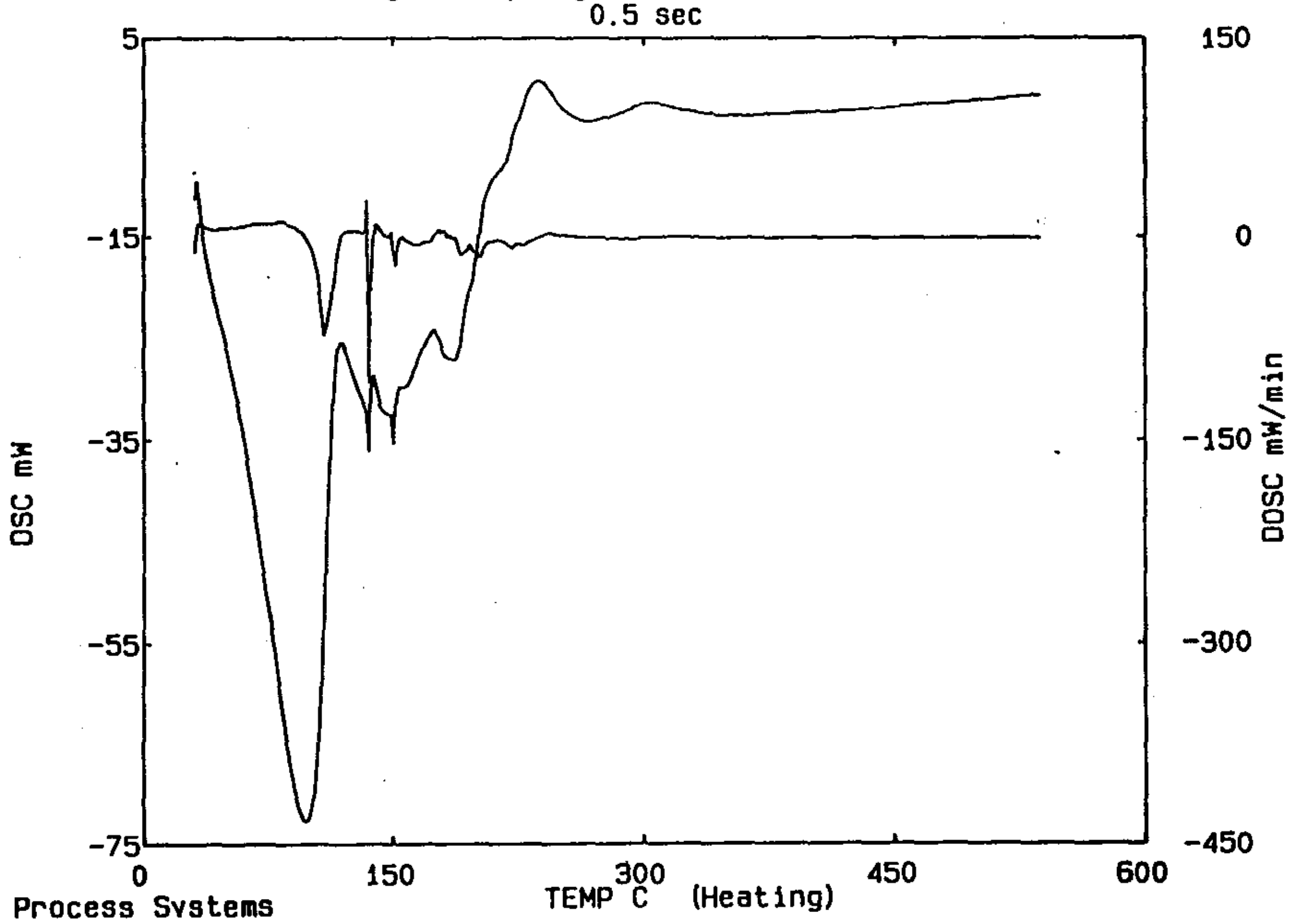
DSC

<Name>
19sept95g
<Date>
95/09/19 14:06

<Sample>
19sept95g
26.977 mg
(26.977 mg)
<Reference>
pt pan
0.000 mg

<Comment>
95-07935
<Sampling>
0.5 sec

<Temp.program [C] [C/min] [min]>
1* 20.0- 550.0 10.00 0.00
<Gas>
nitrogen 300.0 ml/min
0.0 ml/min



Chemical Process Systems

3- 48

WHC-SD-WM-DP-145, REV. 1

TG/DTA

<Name>
19sept95h
<Date>
95/09/19 14:09

<Sample>
19sept95h
29.784 mg
(29.784 mg)
<Reference>
pt pan
0.000 mg

<Comment>
95-07935
<Sampling>
0.5 sec

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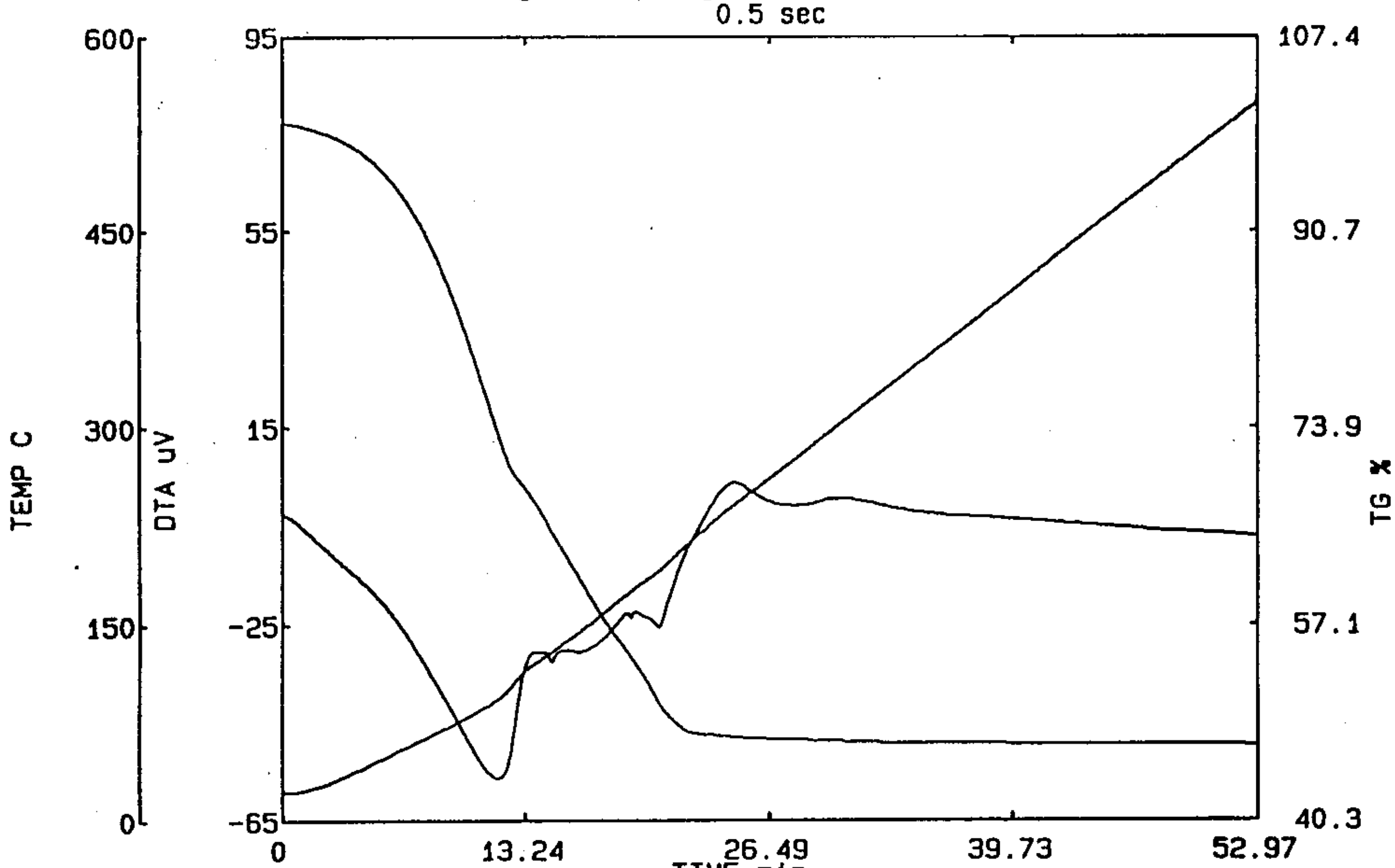
1* 20.0- 550.0 10.00 0.00
<Gas>
nitrogen 300.0 ml/min
0.0 ml/min



Chemical Process Systems

3-49

WHC-SD-WM-DP-145, REV. 1

DSC

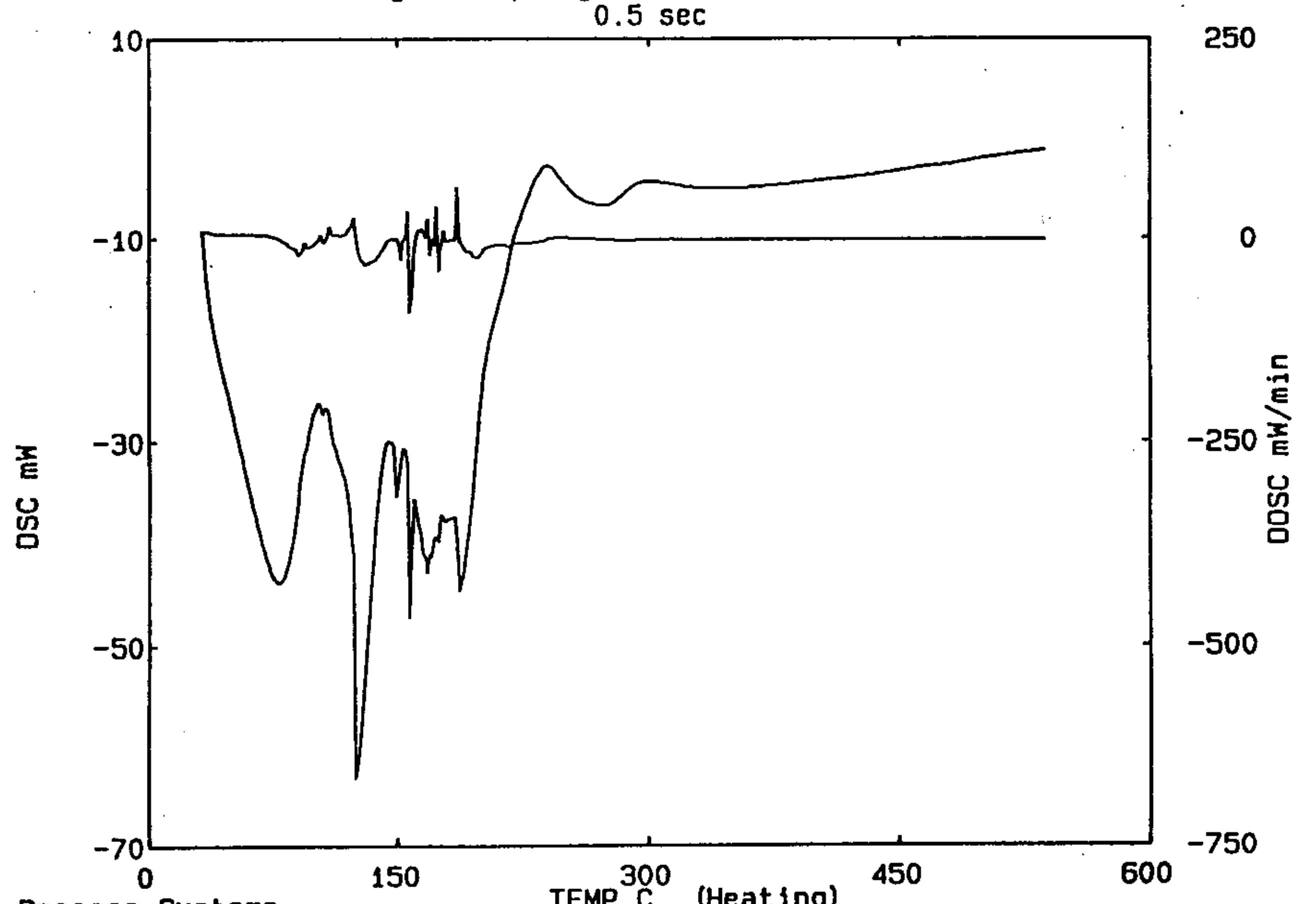
<Name>
19sept95i
<Date>
95/09/19 16:58

<Sample>
19sept95i
24.205 mg
(24.205 mg)
<Reference>
pt pan
0.000 mg

<Comment>
95-07935-2

<Sampling>
0.5 sec

<Temp.program [C] [C/min] [min]>
1* 20.0- 550.0 10.00 0.00
<Gas>
nitrogen 300.0 ml/min
--------------- 0.0 ml/min



Chemical Process Svstems

3-50

WHC-SD-WM-DP-145, REV. 1

WKA

TG/DTA

<Name>
19sept95j

<Date>
95/09/19 17:01

<Sample>
19sept95j
18.818 mg
(18.818 mg)

<Reference>
pt pan
0.000 mg

<Comment>
95-07935-2

<Sampling>
0.5 sec

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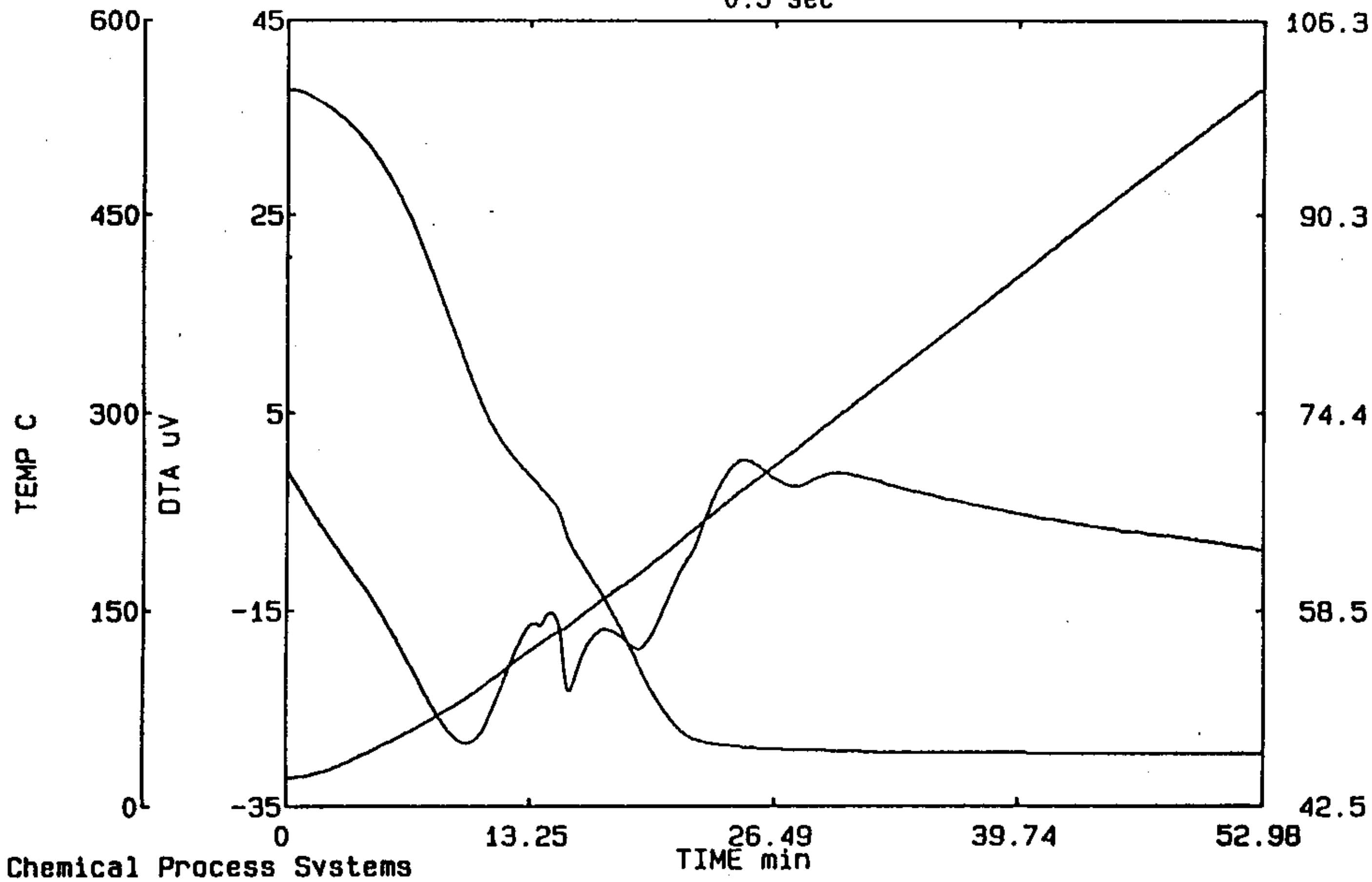
1* 20.0- 550.0 10.00 0.00

<Gas>
nitrogen 300.0 ml/min
---------------- 0.0 ml/min



Chemical Process Systems

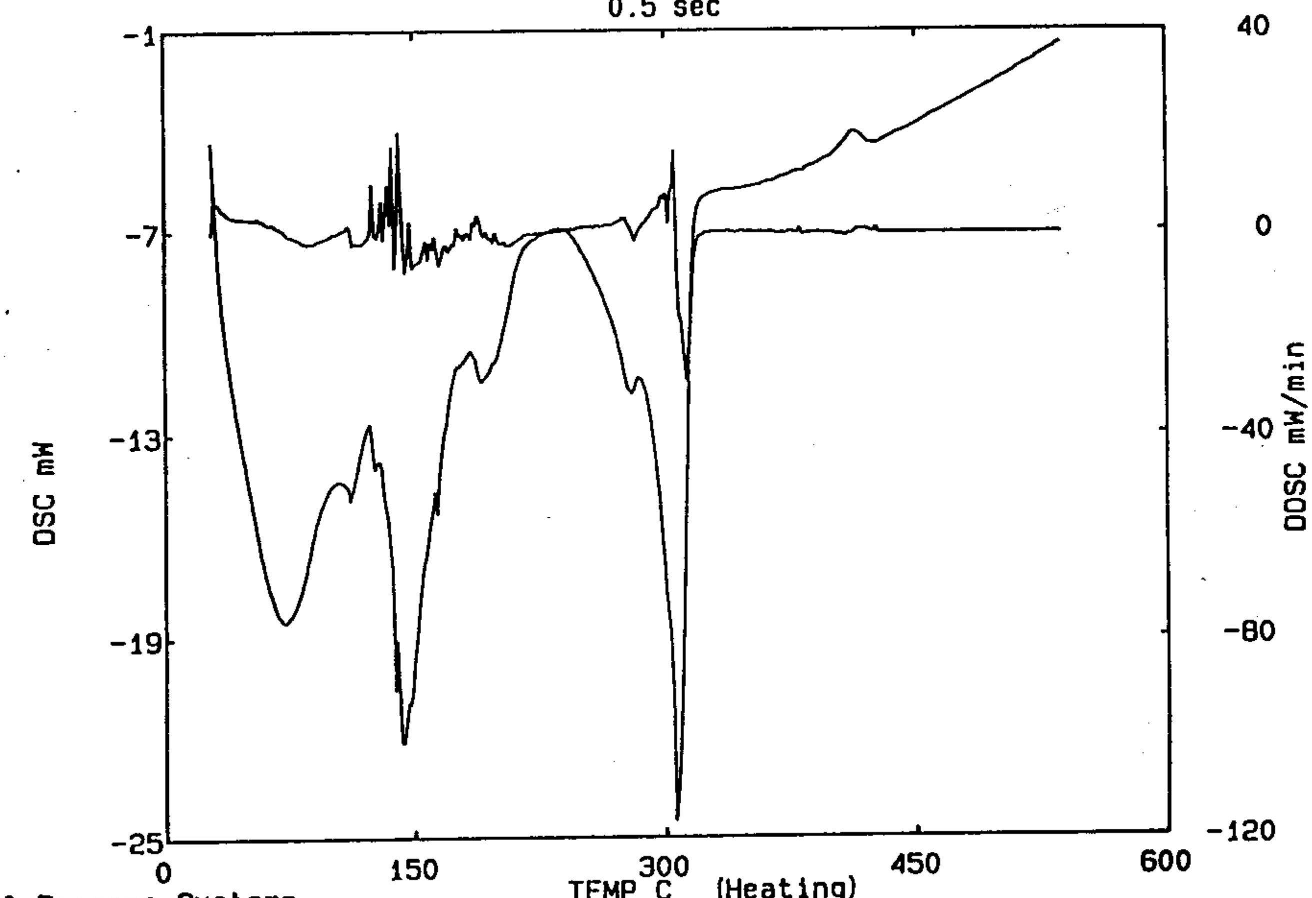
DSC

<Name>
20sept95g
<Date>
95/09/20 12:26

<Sample>
20sept95g
31.843 mg
(31.843 mg)
<Reference>
pt pan
0.000 mg

<Comment>
7316
<Sampling>
0.5 sec

<Temp.program(C) (C/min) (min)>
1* 20.0- 550.0 10.00 0.00
<Gas>
nitrogen 300.0 ml/min
0.0 ml/min



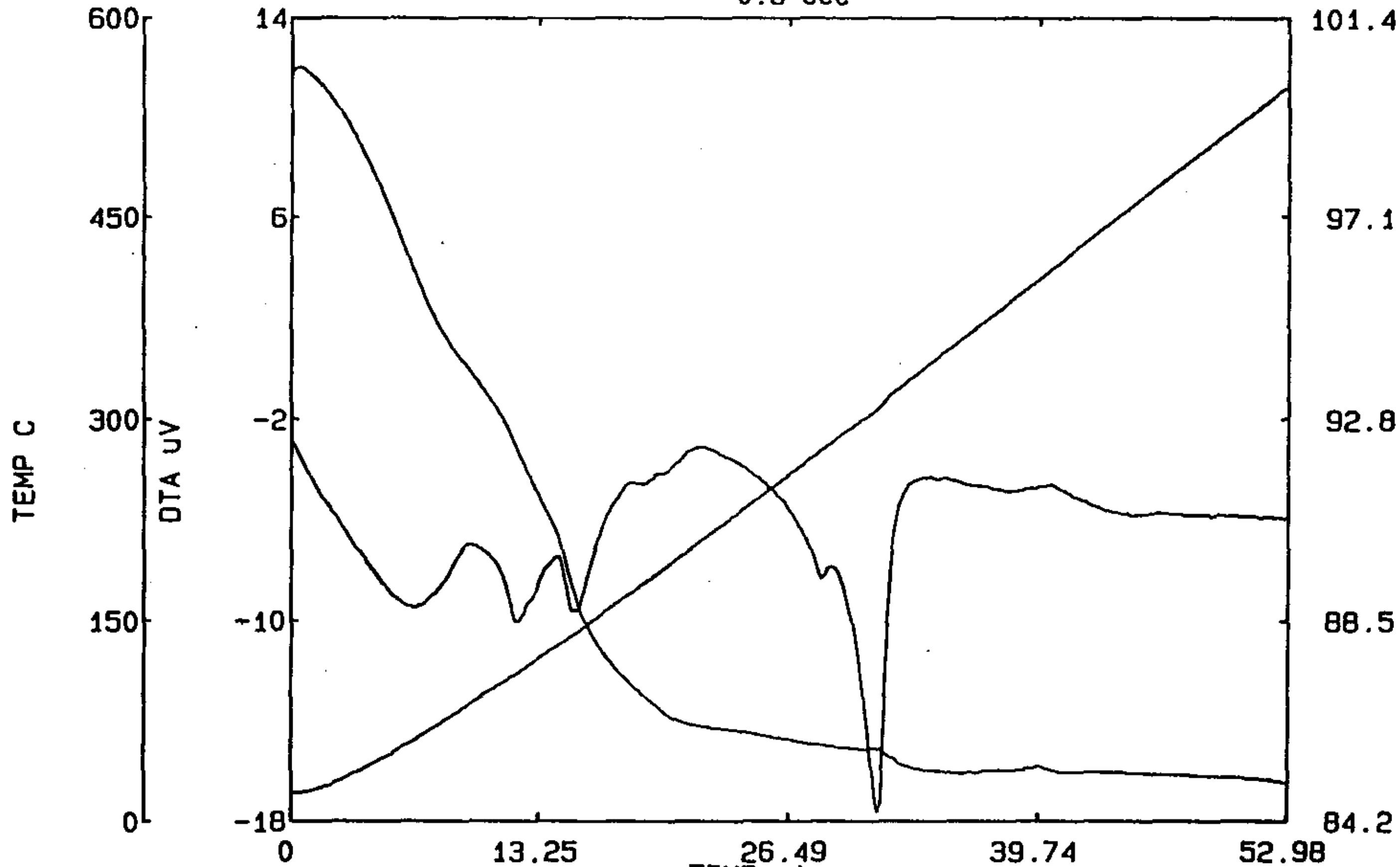
Chemical Process Systems

TG/DTA
<Name>
20sept95h
<Date>
95/09/20 12:29

<Sample>
20sept95h
23.331 mg
(23.331 mg)
<Reference>
pt pan
0.000 mg

<Comment>
7316
<Sampling>
0.5 sec

<Temp.program[C] [C/min] [min]>
1* 20.0- 550.0 10.00 0.00
<Gas>
nitrogen 300.0 ml/min
0.0 ml/min



Chemical Process Systems

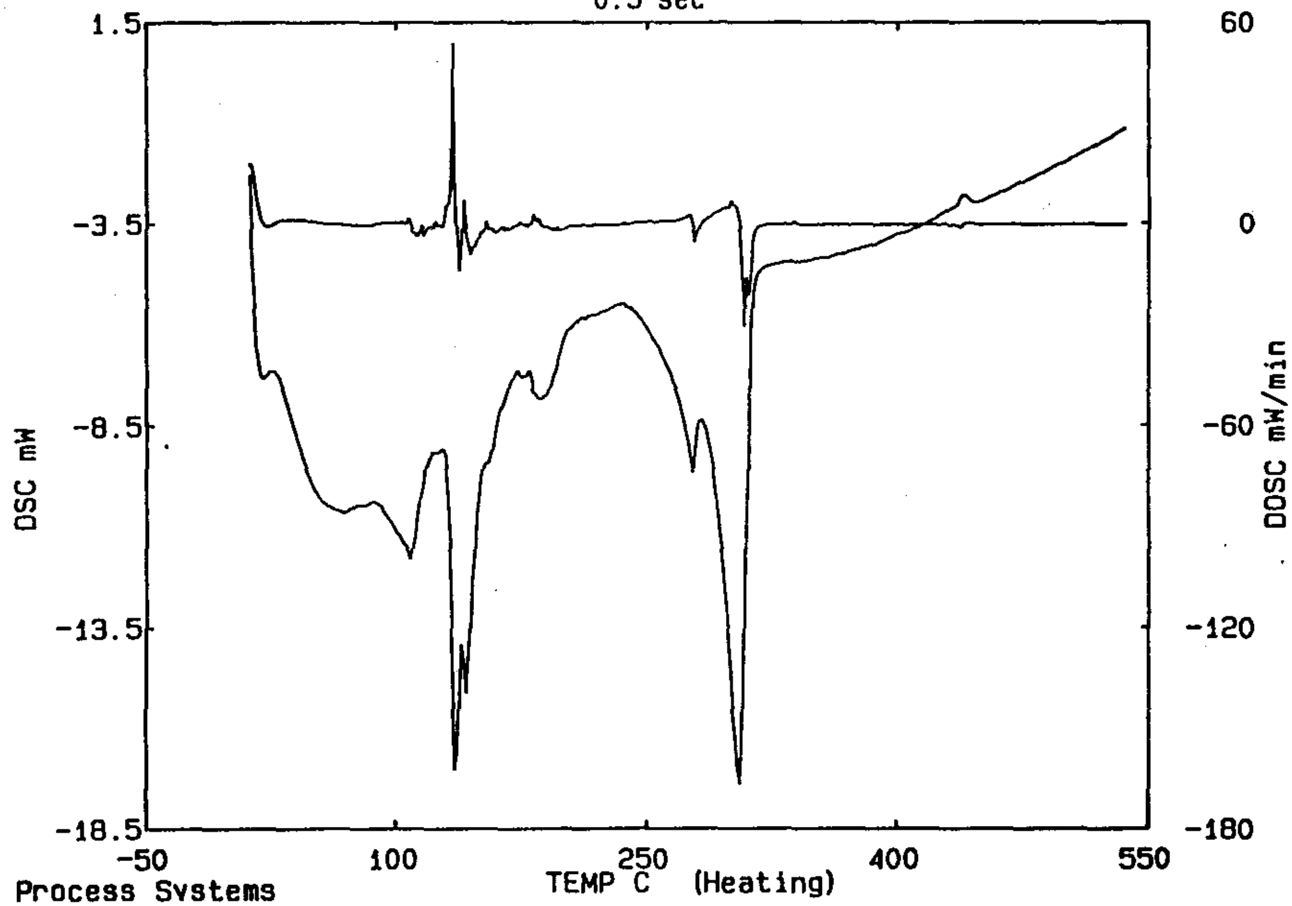
DSC

<Name>
20sept95i
<Date>
95/09/20 13:47

<Sample>
20sept95i
18.382 mg
(18.382 mg)
<Reference>
pt pan
0.000 mg

<Comment>
7316-2
<Sampling>
0.5 sec

<Temp.program [C] [C/min] [min]>
1* 20.0- 550.0 10.00 0.00
<Gas>
nitrogen 300.0 ml/min
0.0 ml/min



Chemical Process Systems

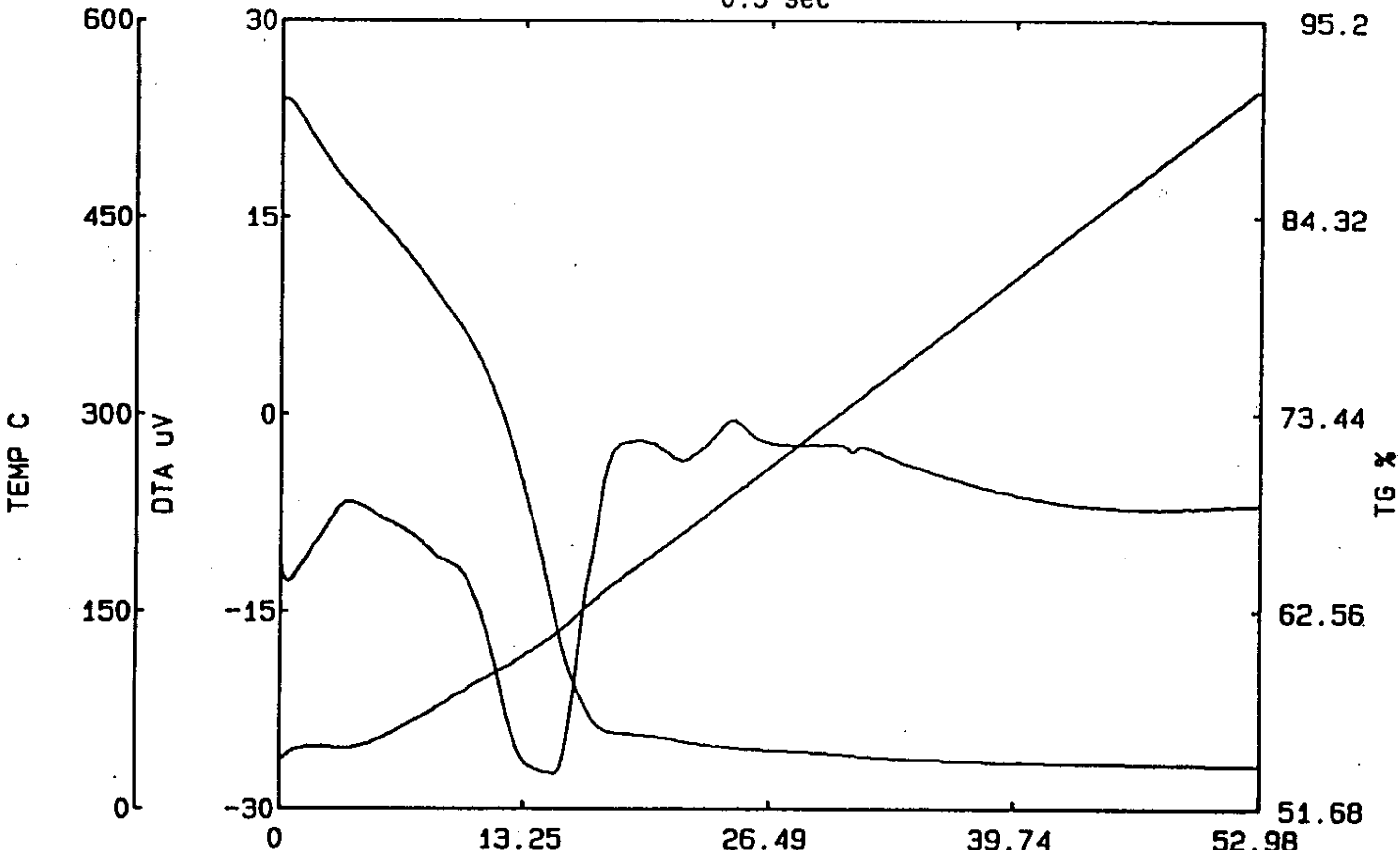
WRa

TG/DTA
<Name>
20sept95j
<Date>
95/09/20 13:49

<Sample>
20sept95j
18.382 mg
(18.382 mg)
<Reference>
pt pan
0.000 mg

<Comment>
7316-2
<Sampling>
0.5 sec

<Temp.program [C] [C/min] [min]>
1* 20.0- 550.0 10.00 0.00
<Gas>
nitrogen 300.0 ml/min
0.0 ml/min



Chemical Process Systems

3-55

WHC-SD-WM-DP-145, REV. 1

DSC

<Name>
15sept95a

<Date>
95/09/15 06:31

<Sample>
15sept95a
7.391 mg
(7.391 mg)

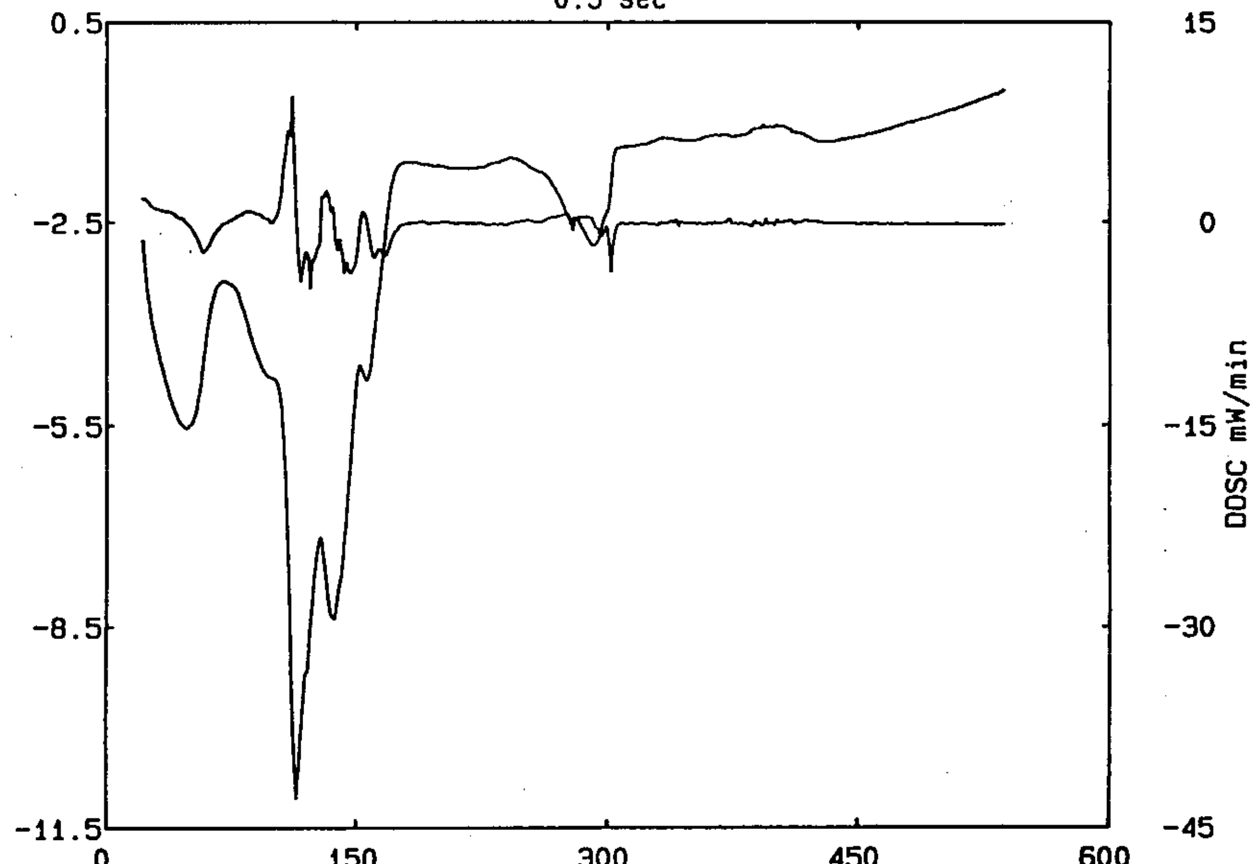
<Reference>
pt pan
0.000 mg

<Comment>
7322

<Sampling>
0.5 sec

<Temp.program [C] [C/min] [min]>
1* 20.0- 550.0 10.00 0.00

<Gas>
nitrogen 300.0 ml/min
0.0 ml/min



Chemical Process Systems

WHC-SD-WM-DP-145, REV. 1

3-56

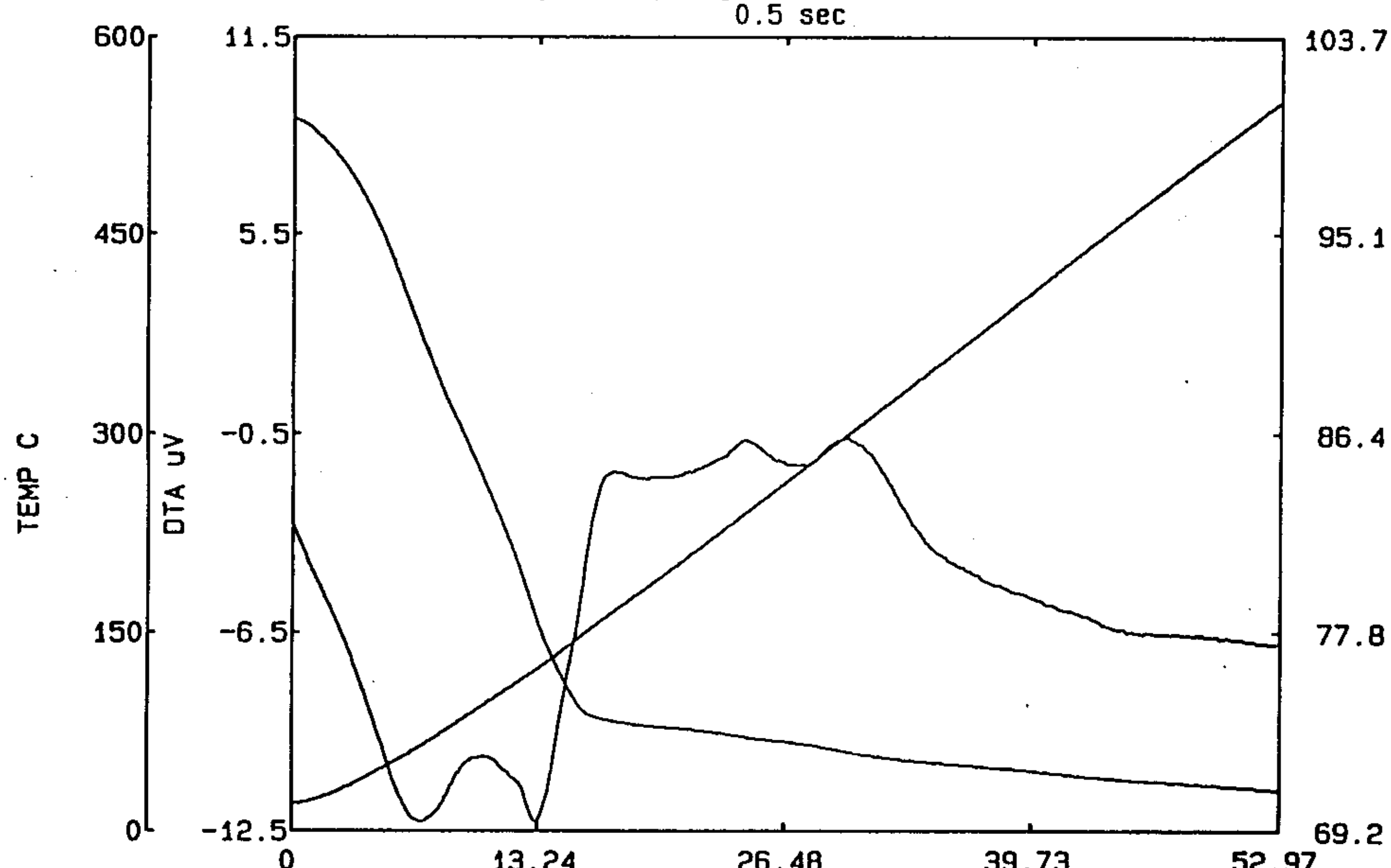
TG/DTA

<Name>
15sept95b
<Date>
95/09/15 06:36

<Sample>
15sept95b
17.351 mg
(17.351 mg)
<Reference>
pt pan
0.000 mg

<Comment>
7322
<Sampling>
0.5 sec

<Temp.program [C] [C/min] [min]>
1* 20.0- 550.0 10.00 0.00
<Gas>
nitrogen 300.0 ml/min
0.0 ml/min



Chemical Process Systems

3-57

WHC-SD-WM-DP-145, REV. 1

DSC

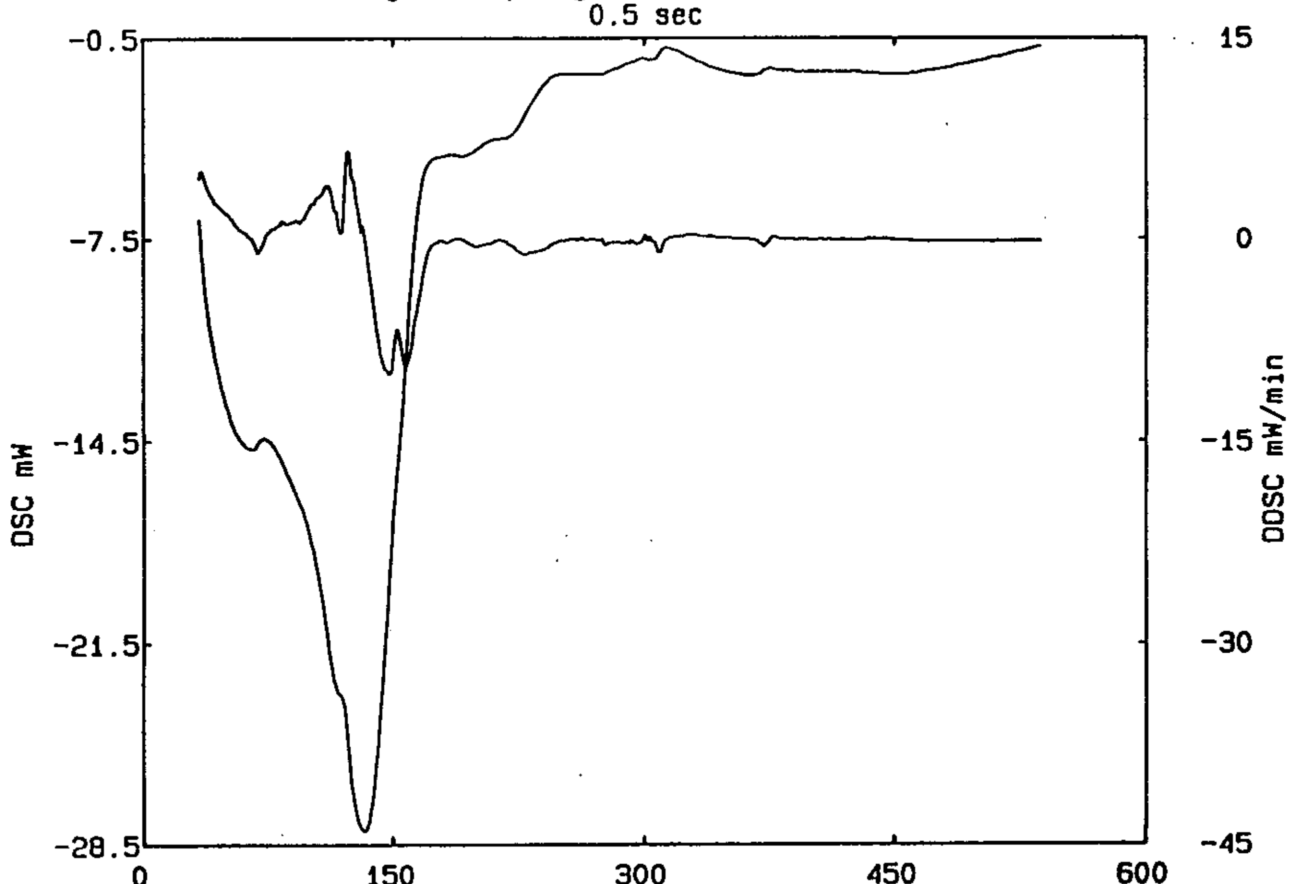
<Name>
19sept95e
<Date>
95/09/19 11:35

<Sample>
19sept95e
18.071 mg
(18.071 mg)
<Reference>
pt pan
0.000 mg

<Comment>
7322-2

<Sampling>
0.5 sec

<Temp.program[C] [C/min] [min]>
1* 20.0- 550.0 10.00 0.00
<Gas>
nitrogen 300.0 ml/min
---------------- 0.0 ml/min



Chemical Process Systems

3-58

WHC-SD-WM-DP-145, REV. 1

TG/DTA

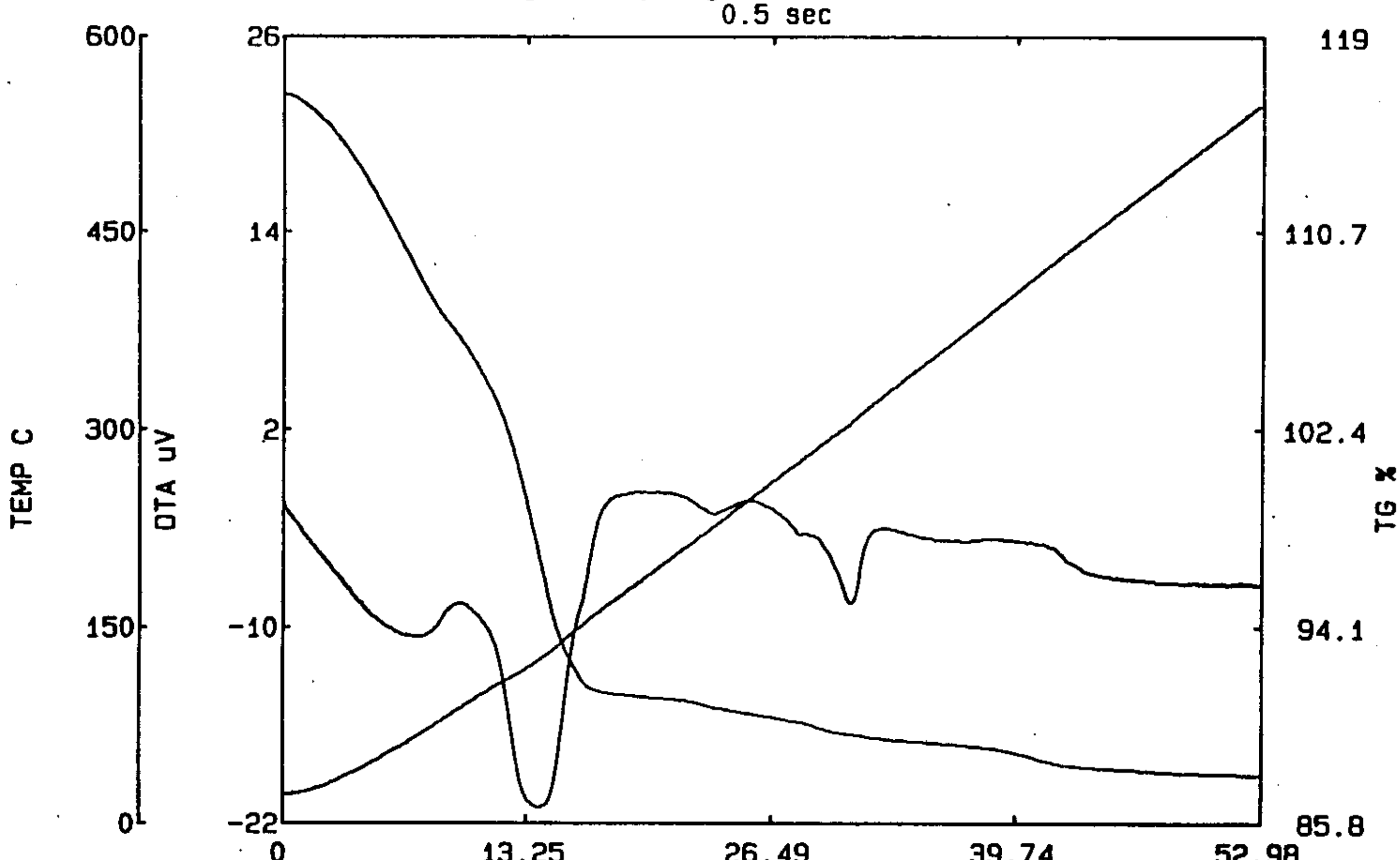
<Name>
19sept95f
<Date>
95/09/19 11:38

<Sample>
19sept95f
18.071 mg
(18.071 mg)
<Reference>
pt pan
0.000 mg

<Comment>
7322-2

<Sampling>
0.5 sec

<Temp.program[C] [C/min] [min]>
1* 20.0- 550.0 10.00 0.00
<Gas>
nitrogen 300.0 ml/min
--------------- 0.0 ml/min



Chemical Process Systems

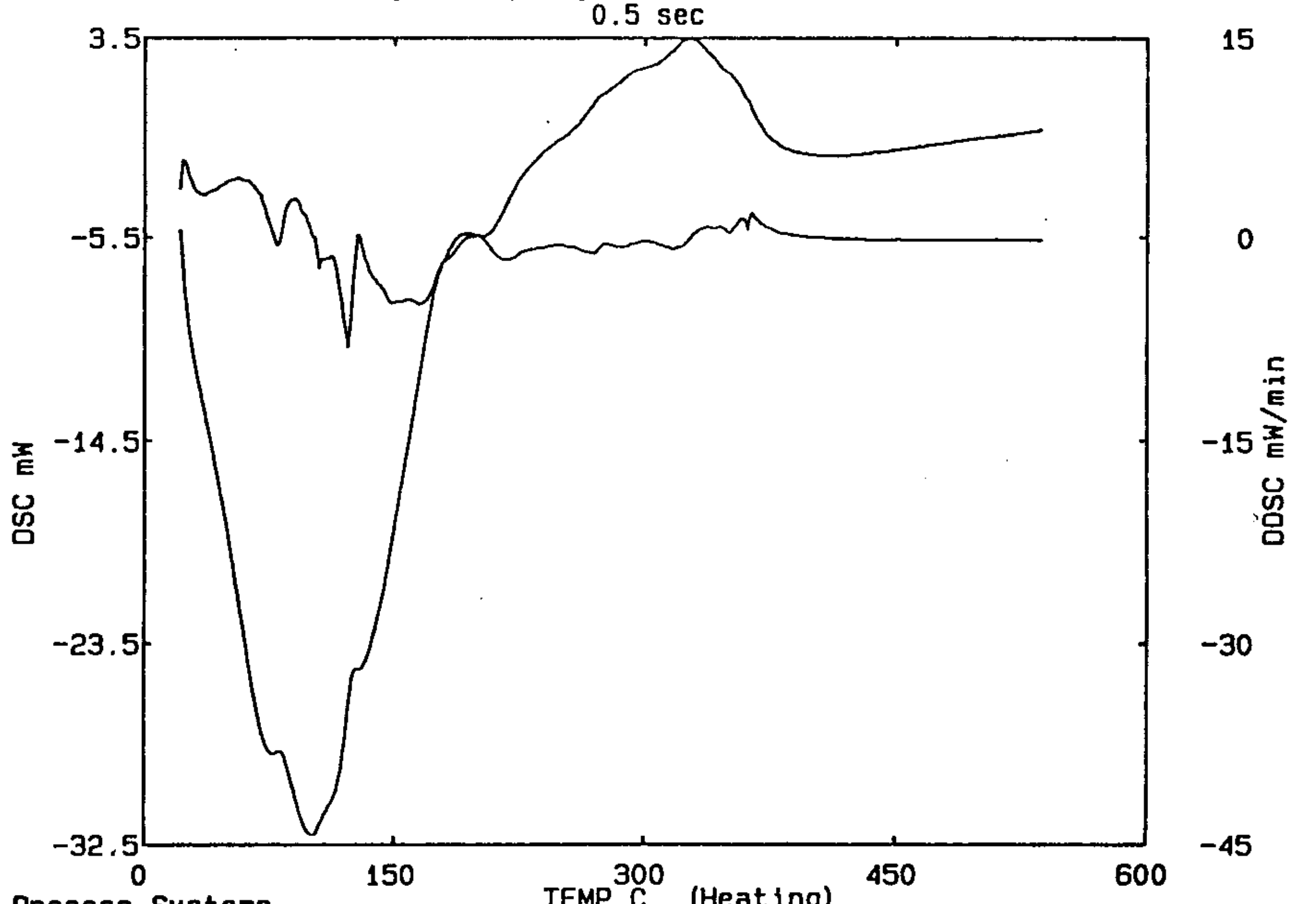
DSC

<Name>
19sept95a
<Date>
95/09/19 06:52

<Sample>
19sept95a
23.424 mg
(23.424 mg)
<Reference>
pt pan
0.000 mg

<Comment>
7319
<Sampling>
0.5 sec

<Temp.program [C] [C/min] [min]>
1* 20.0- 550.0 10.00 0.00
<Gas>
nitrogen 300.0 ml/min
0.0 ml/min



Chemical Process Systems

3-60

WHC-SD-WM-DP-145, REV. 1

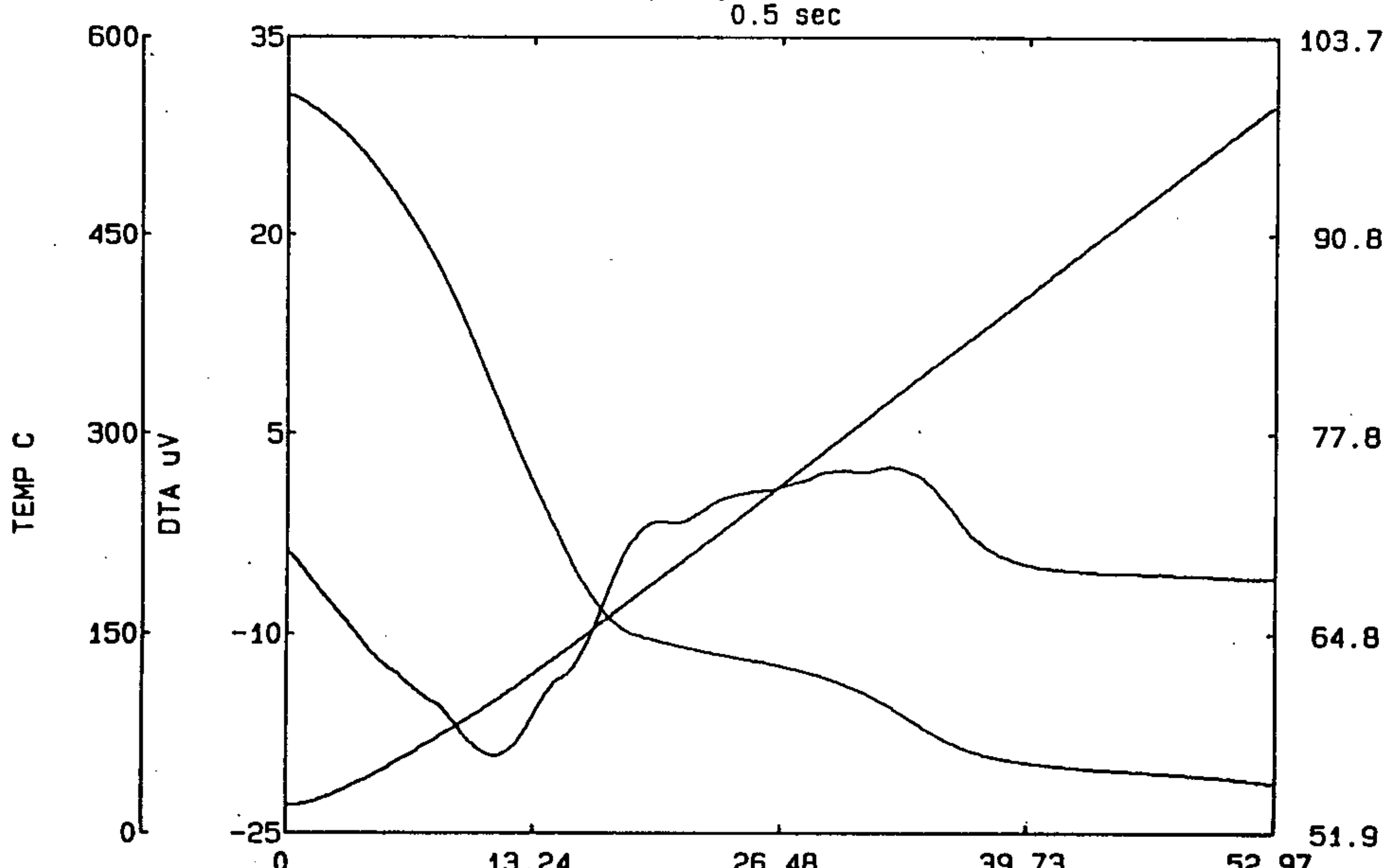
TG/DTA

<Name>
19sept95b
<Date>
95/09/19 06:55

<Sample>
19sept95b
19.283 mg
(19.283 mg)
<Reference>
pt pan
0.000 mg

<Comment>
7319
<Sampling>
0.5 sec

<Temp.program [C] [C/min] [min]>
1* 20.0- 550.0 10.00 0.00
<Gas>
nitrogen 300.0 ml/min
0.0 ml/min



Chemical Process Systems

3- 61

WHC-SD-WM-DP-145, REV. 1

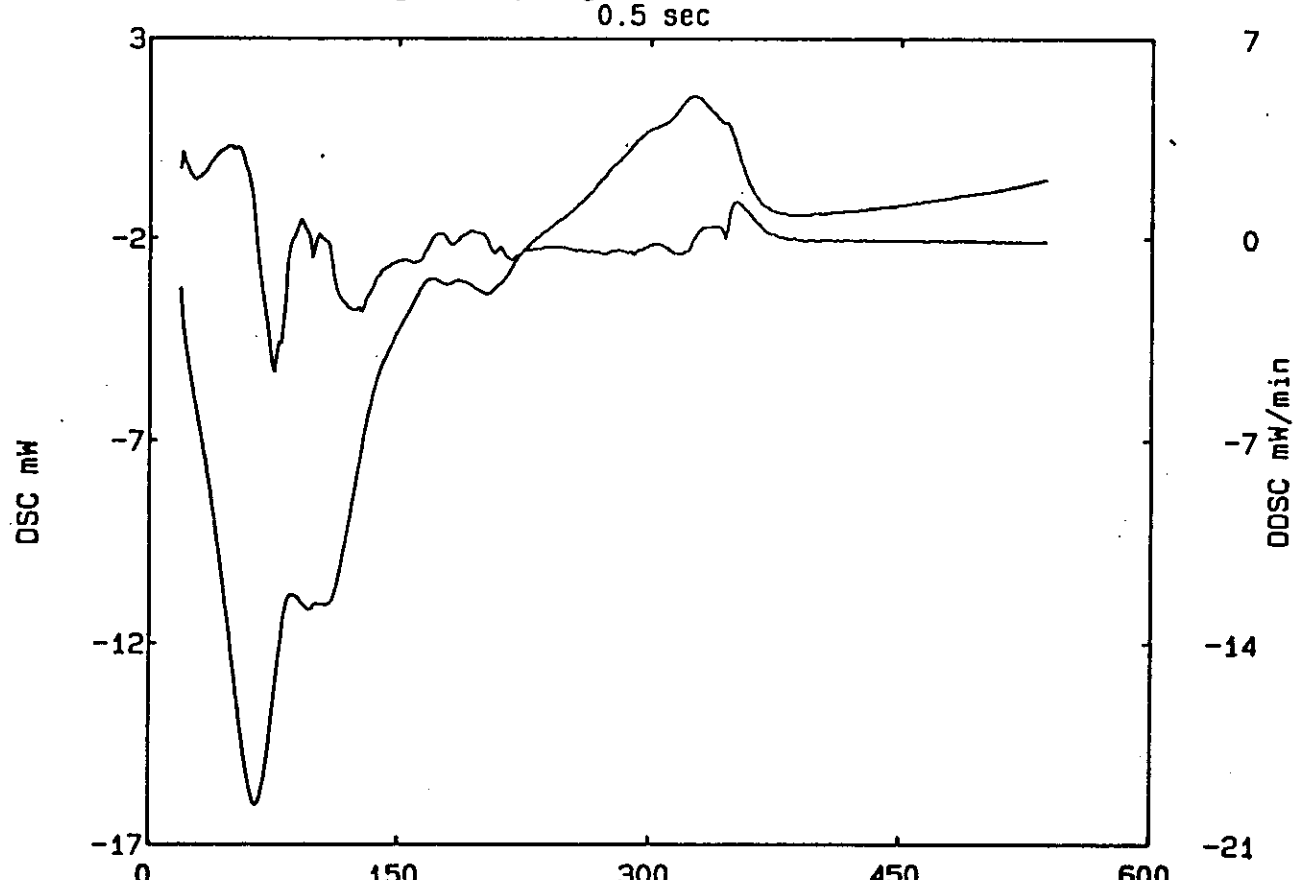
DSC

<Name>
19sept95c
<Date>
95/09/19 08:57

<Sample>
19sept95c
12.027 mg
(12.027 mg)
<Reference>
pt pan
0.000 mg

<Comment>
7319-2
<Sampling>
0.5 sec

<Temp.program[C] [C/min] [min]>
1* 20.0- 550.0 10.00 0.00
<Gas>
nitrogen 300.0 ml/min
0.0 ml/min



Chemical Process Systems

TEMP C (Heating)

WHC-SD-WM-DP-145, REV 1

3-62

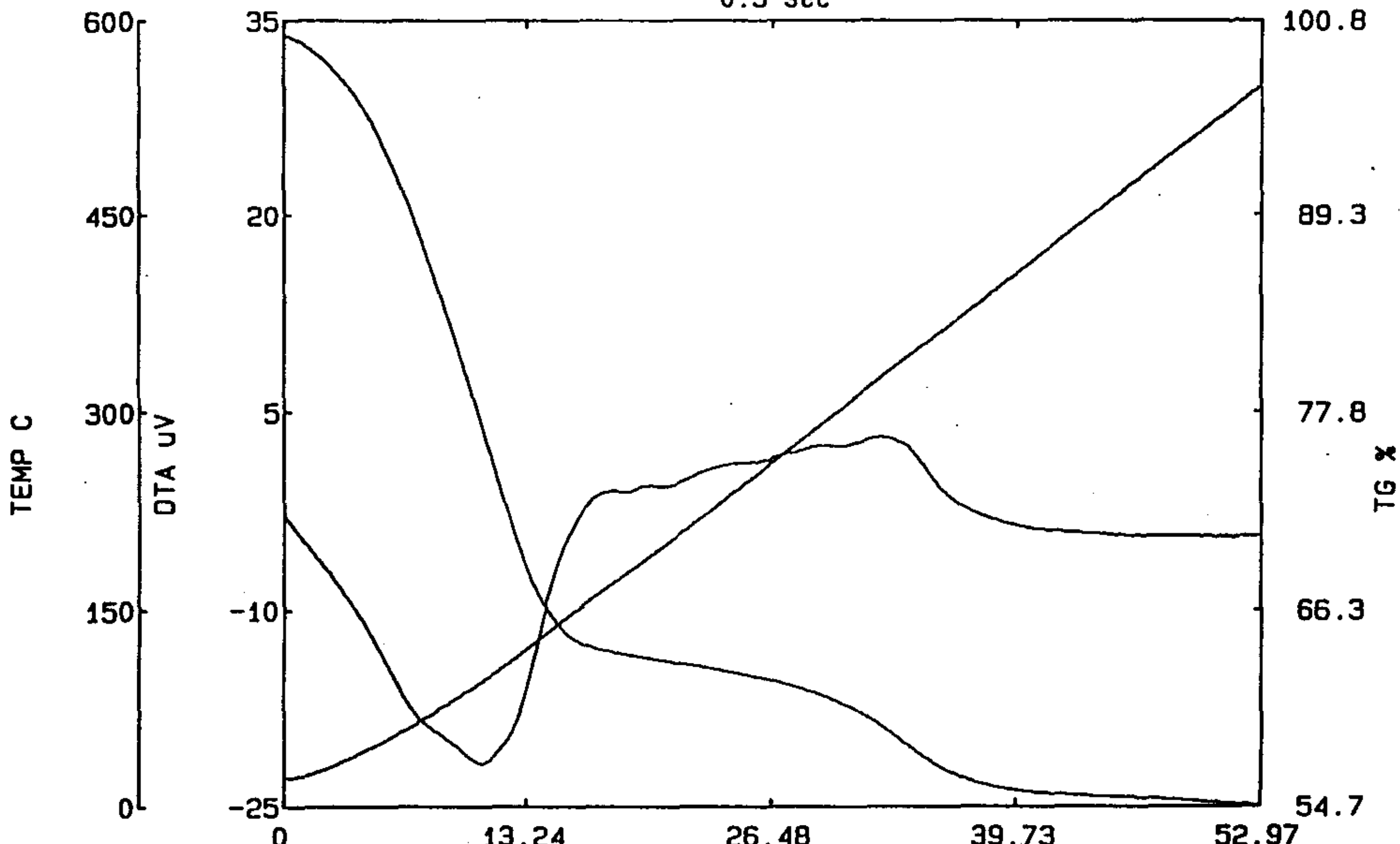
TG/DTA

<Name>
19sept95d
<Date>
95/09/19 08:59

<Sample>
19sept95d
17.353 mg
(17.353 mg)
<Reference>
pt pan
0.000 mg

<Comment>
7319-2
<Sampling>
0.5 sec

<Temp.program[C] [C/min] [min]>
1* 20.0- 550.0 10.00 0.00
<Gas>
nitrogen 300.0 ml/min
0.0 ml/min



Chemical Process Systems

DSC

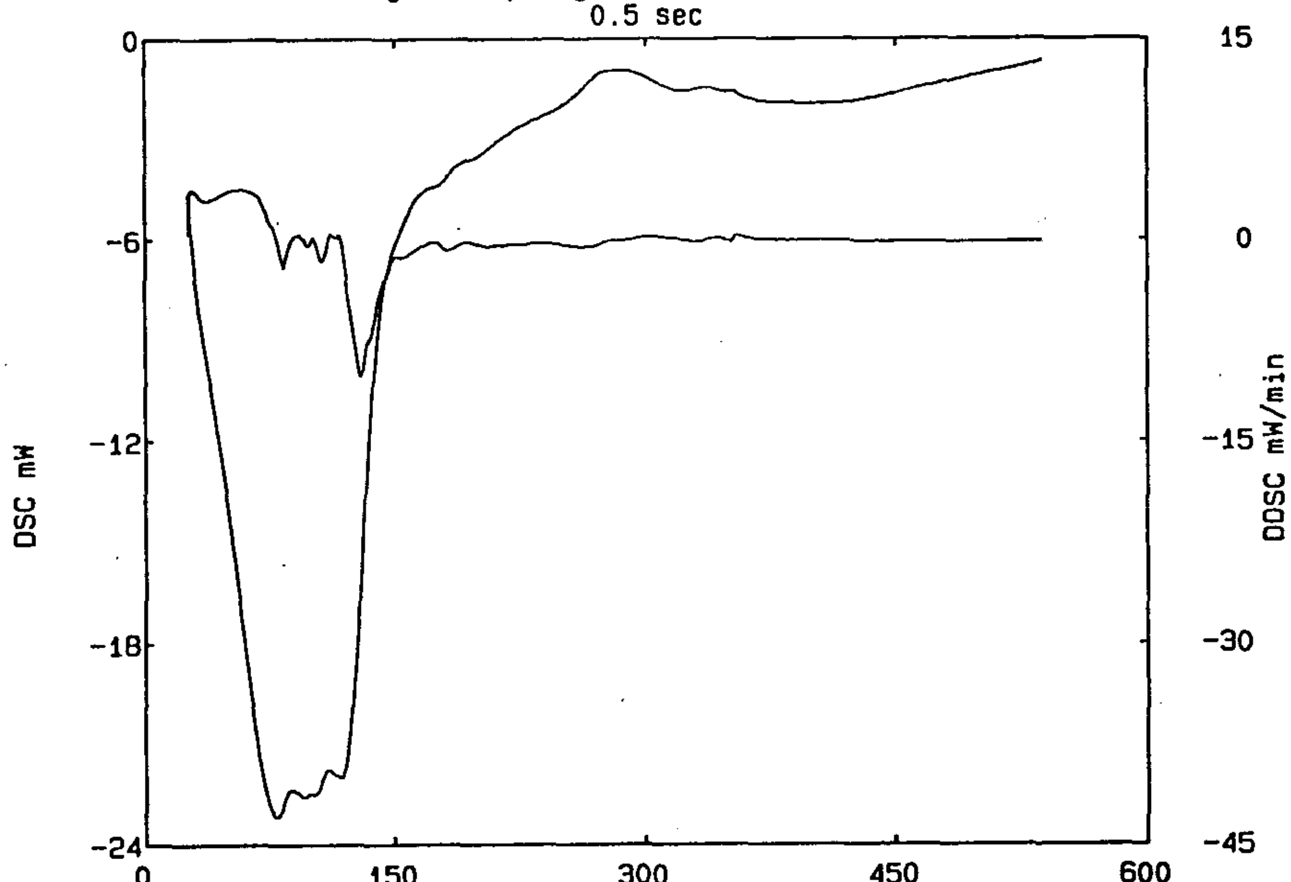
<Name>
18sept95e
<Date>
95/09/18 17:33

<Sample>
18sept95e
15.000 mg
(15.000 mg)
<Reference>
pt pan
0.000 mg

<Comment>
7318

<Sampling>
0.5 sec

<Temp.program[C] [C/min] [min]>
1* 20.0- 550.0 10.00 0.00
<Gas>
nitrogen 300.0 ml/min
--------------- 0.0 ml/min



Chemical Process Systems

TG/DTA <Name> 18sept95f <Date> 95/09/18 17:35

<Sample> 18sept95f 11.944 mg (11.944 mg) <Reference> pt pan 0.000 mg

<Comment> 7318

<Sampling> 0.5 sec

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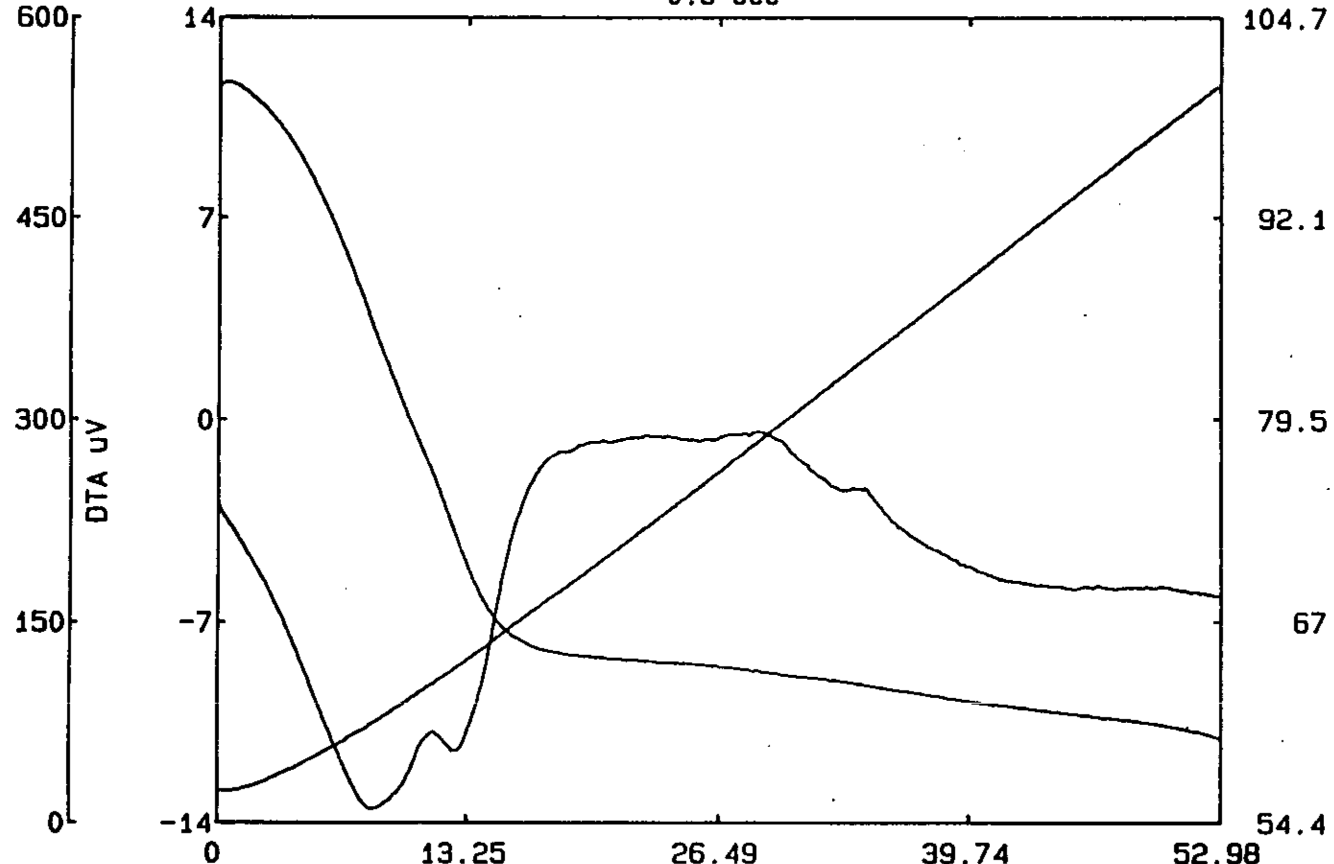
<Gas> nitrogen 300.0 ml/min 0.0 ml/min

WHC-SD-WM-DP-145, REV. 1

I-50



Chemical Process Systems

3-65

WHQ

WHC-SD-WM-DP-145, REV. 1

DSC

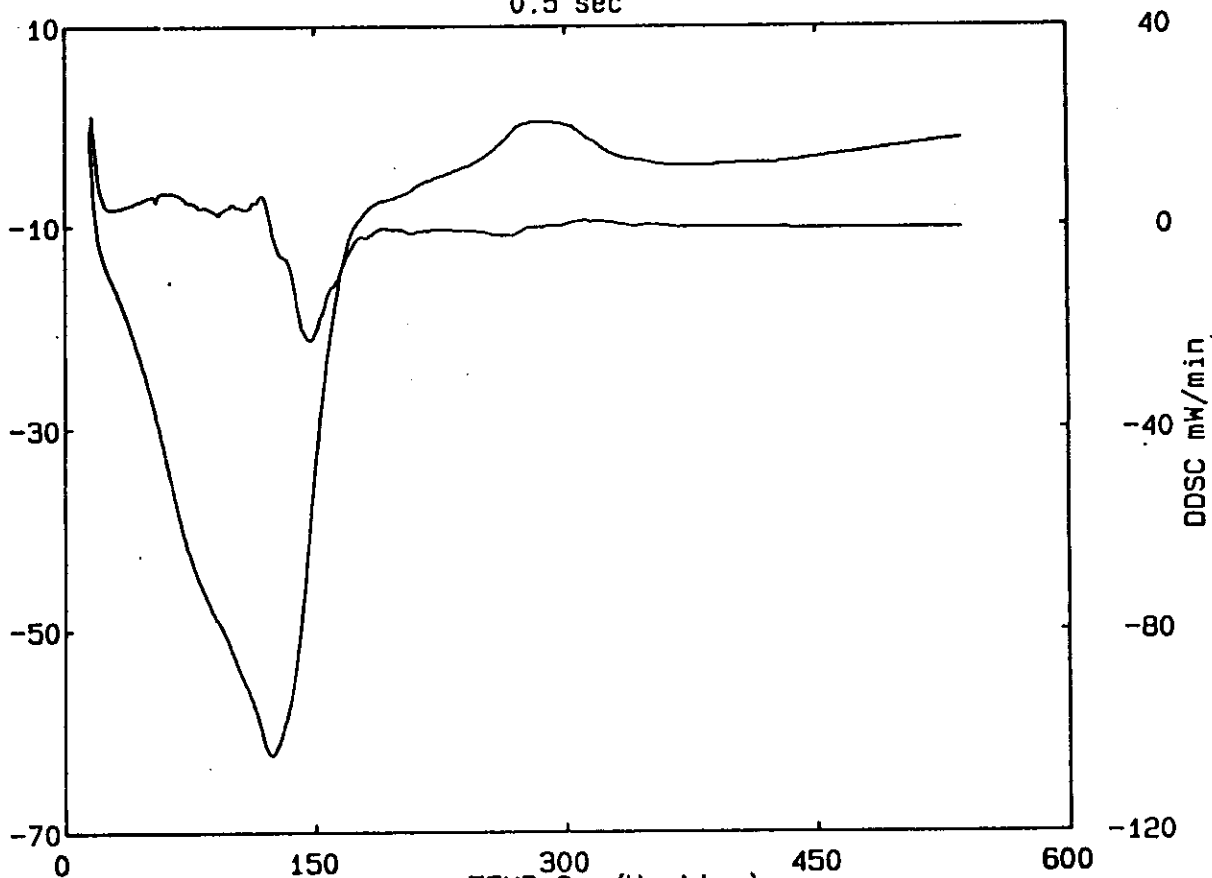
<Name>
18sept95g
<Date>
95/09/18 19:31

<Sample>
18sept95g
36.006 mg
(36.006 mg)
<Reference>
pt pan
0.000 mg

<Comment>
7318-2

<Sampling>
0.5 sec

<Temp.program [C] [C/min] [min]>
1* 20.0- 550.0 10.00 0.00
<Gas>
nitrogen 300.0 ml/min
---------------- 0.0 ml/min



Chemical Process Systems

3-66

TG/DTA

<Name>
18sept95h
<Date>
95/09/18 19:34

<Sample>
18sept95h
26.051 mg
(26.051 mg)
<Reference>
pt pan
0.000 mg

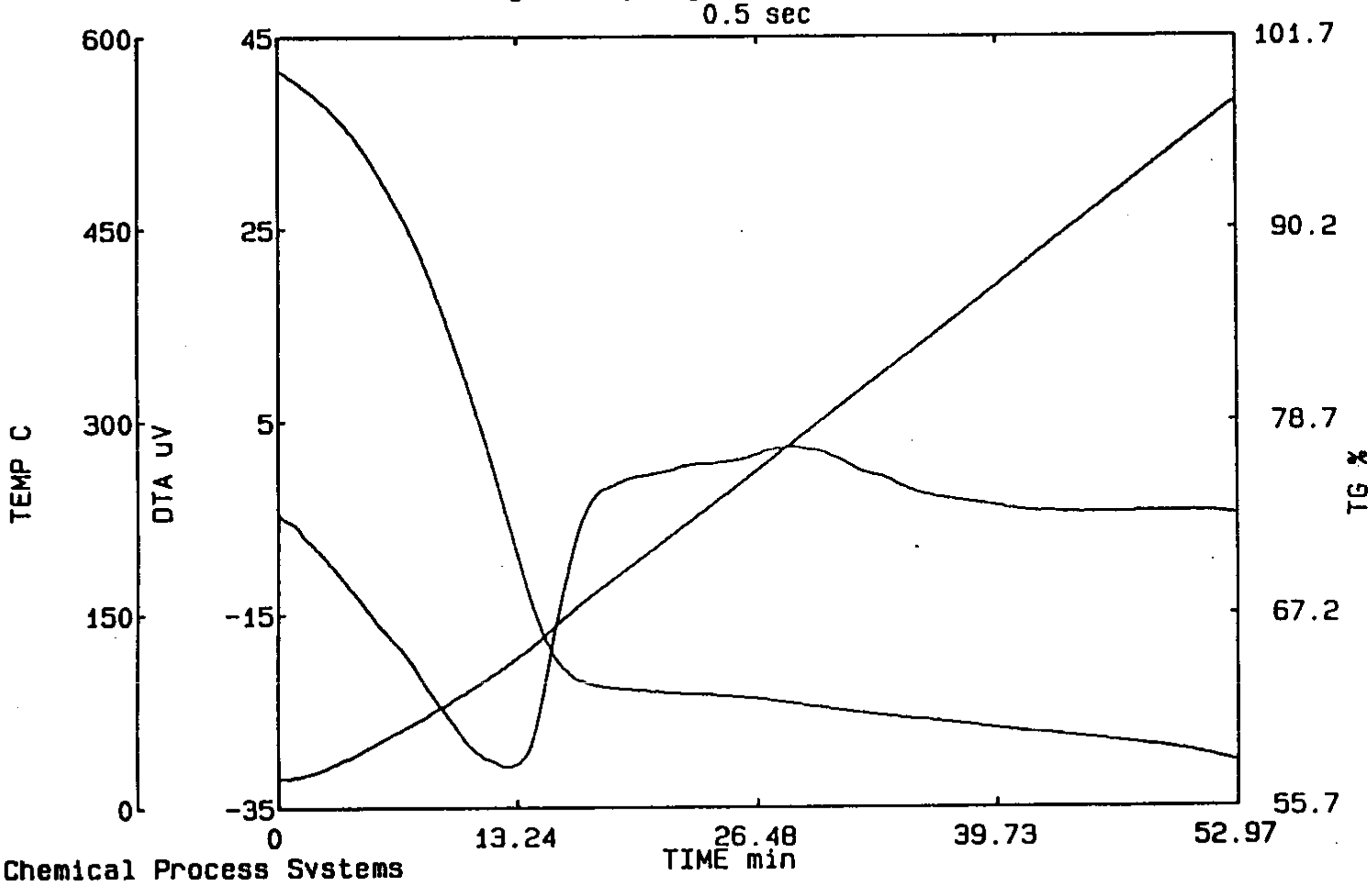
<Comment>
7318-2

<Sampling>
0.5 sec

<Temp.program [C] [C/min] [min]>
1* 20.0- 550.0 10.00 0.00
<Gas>
nitrogen 300.0 ml/min
---------------- 0.0 ml/min

WHC-SD-WM-DP-145, REV. 1

1-52



Chemical Process Systems

3-67

WRA

DSC

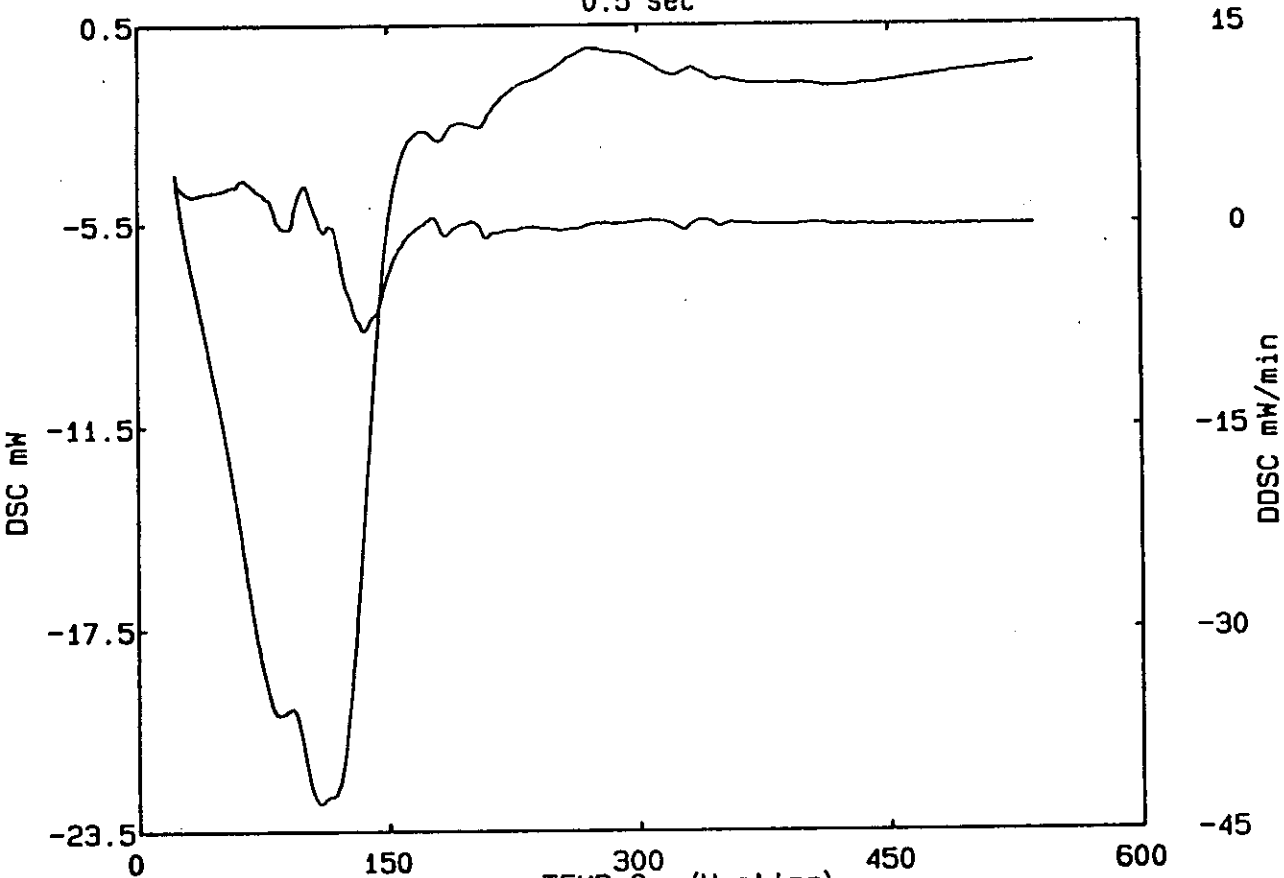
<Name>
18sept95a
<Date>
95/09/18 09:46

<Sample>
18sept95a
13.397 mg
(13.397 mg)
<Reference>
pt pan
0.000 mg

<Comment>
7317

<Sampling>
0.5 sec

<Temp.program [C] [C/min] [min]>
1* 20.0- 550.0 10.00 0.00
<Gas>
nitrogen 300.0 ml/min
0.0 ml/min



Chemical Process Systems

WHA

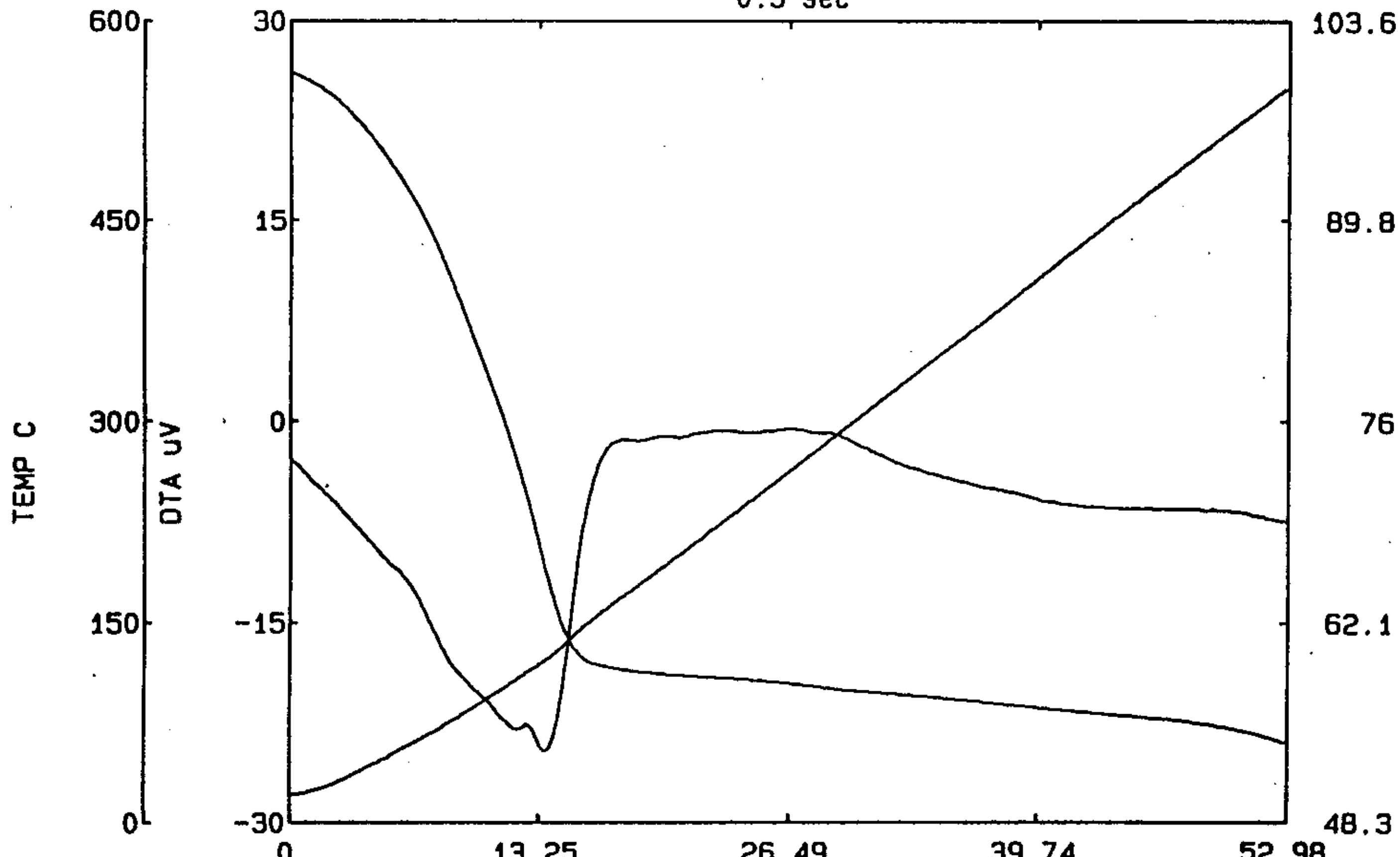
TG/DTA
<Name>
18sept95b
<Date>
95/09/18 09:49

<Sample>
18sept95b
14.483 mg
(14.483 mg)
<Reference>
pt pan
0.000 mg

<Comment>
7317

<Sampling>
0.5 sec

<Temp.program(C) (C/min) (min)>
1* 20.0- 550.0 10.00 0.00
<Gas>
nitrogen 300.0 ml/min
--------------- 0.0 ml/min



Chemical Process Systems

DSC

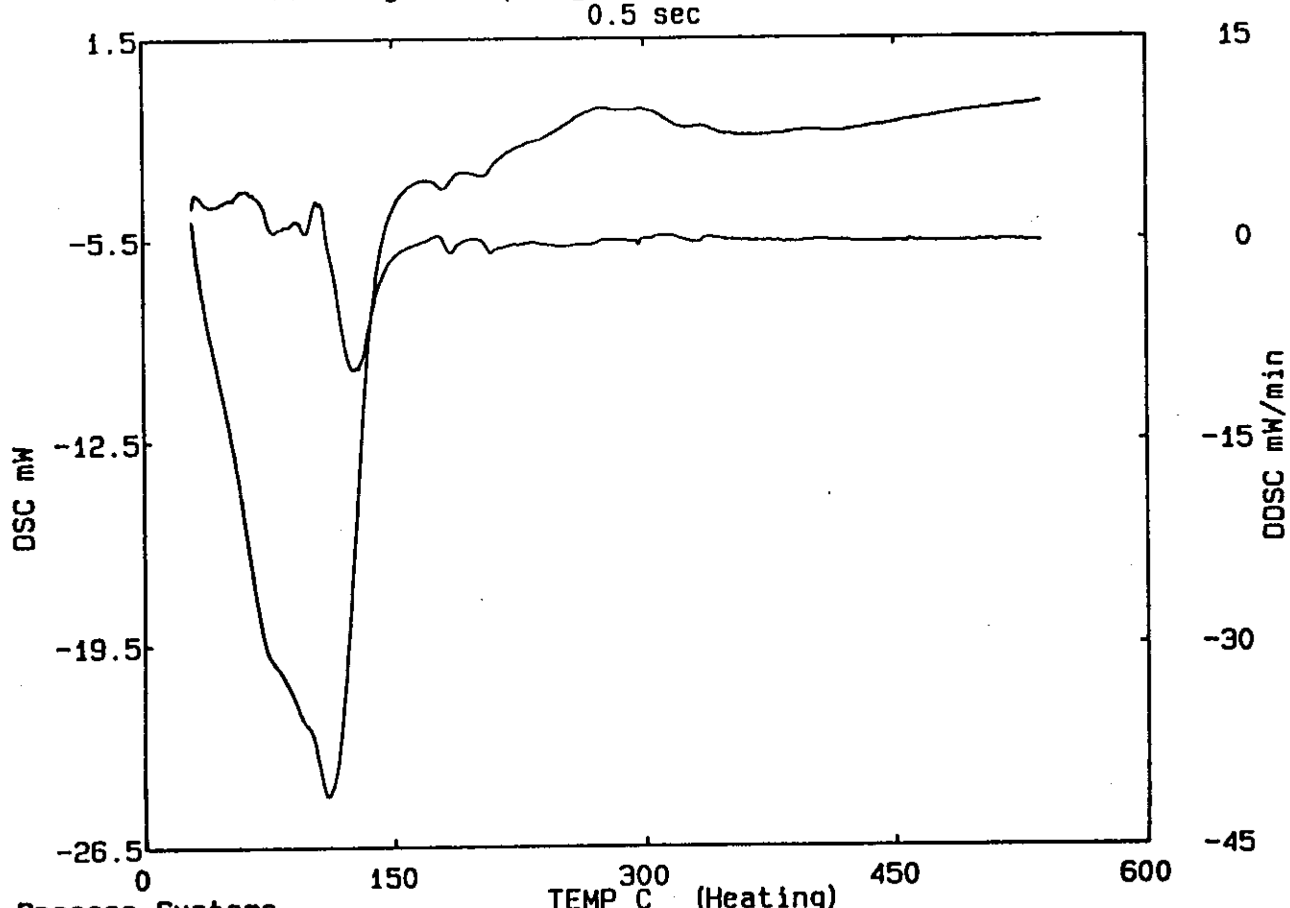
<Name>
18sept95c
<Date>
95/09/18 13:16

<Sample>
18sept95c
13.697 mg
(13.697 mg)
<Reference>
pt pan
0.000 mg

<Comment>
7317-2

<Sampling>
0.5 sec

<Temp.program [C] [C/min] [min]>
1* 20.0- 550.0 10.00 0.00
<Gas>
nitrogen 300.0 ml/min
--------------- 0.0 ml/min



Chemical Process Systems

3-70

WHC-SD-WM-DP-145, REV. 1

TG/DTA

<Name>
18sept95d

<Date>
95/09/18 13:19

<Sample>
18sept95d
36.730 mg
(36.730 mg)

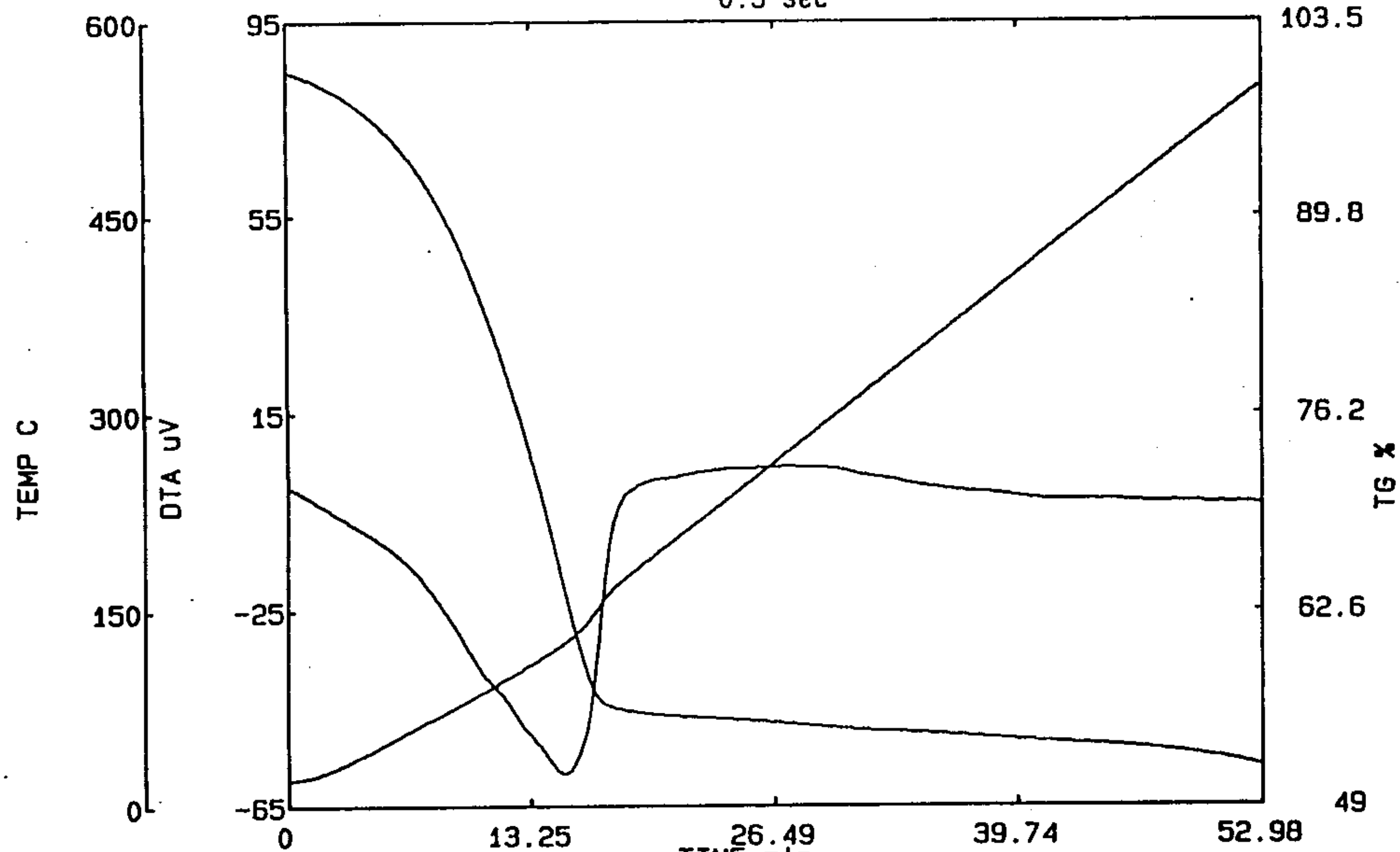
<Reference>
pt pan
0.000 mg

<Comment>
7317-2

<Sampling>
0.5 sec

<Temp.program [C] [C/min] [min]>
1* 20.0- 550.0 10.00 0.00

<Gas>
nitrogen 300.0 ml/min
0.0 ml/min



Chemical Process Systems

3-71

WHC-SD-WM-DP-145, REV. 1

62

Density

The density of each sample with the exception of Quarter Segment 2A was measured using a displacement method. Due to the limited sample available for these analyses, only a single measurement was made for each sample. The density of Segment 2A was not measured because sufficient sample was not available to perform an accurate density measurement. The density methods described in PNL technical procedure PNL-ALO-501 could not be followed because of the limited sample available. The results of density measurements are given in Table 1-8.

Table 1-8: Tank BY-108, Core 99 Density Results

WHC Sample Number	Segment ID	Density (g/mL)
7313	1	1.59
7314	2D	1.36
7397	2 Drainable Liquid	1.21
7316	3A	1.57
7398	3 Drainable Liquid	1.21
7322	4A	1.51
7319	4B	1.51
7318	4C	1.53
7317	4D	1.55

Experimental Procedure

The density of the solid samples was obtained by a displacement method. The samples were placed in preweighed graduated cylinder with a known volume and mass of mineral oil. After the sample was placed in the mineral oil, the volume and mass of the sample and oil were measured. The density of the sample was then calculated from the sample mass and sample volume obtained from subtracting the mass and volume of the mineral oil from the mass and volume of the sample plus mineral oil. The density of the drainable liquid was obtained by accurately measuring the mass of the liquid pipetted from a fixed volume pipet.

SECTION 2

INORGANIC CHEMISTRY

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Inductively Coupled Plasma (ICP) Spectrometry Analysis

Sample Preparation and Analysis:

ICP analyses were performed on fusions prepared from homogenized material from Core 99 Segment 1 and Quarter Segments 2A, 2D, 3A, and 4A through 4D. The samples were prepared following procedures PNL-ALO-114 "Solubilization of Metals from Solids Using a Na_2O_2-NaOH Fusion" and PNL-ALO-115 "Solubilization of Metals from Solids Using a KOH-KNO_3 Fusion." Also, ICP analyses were performed on acid digestions of the drainable liquids from Segment 2 and Segment 3. The drainable liquids samples were prepared following procedure PNL-ALO-128 "HNO_3-HCl Acid Extraction of Liquids for Metals Analysis Using a Dry-block Heater." The core material fusions and drainable liquid acid digestions were performed in the Shielded Analytical Laboratory (SAL) with sample solution aliquots being transferred to the Inorganic Analysis Group for ICP analysis. All ICP metals analyses were performed on a Jarrell-Ash ICP 800A system following procedure PNL-ALO-211, "Determination of Elements by Inductively Coupled Argon Plasma Atomic Emission Spectrometry".

Analytical Results:

The KOH-KNO_3 fusion analytical results are presented in Tables 2.1a through 2.1e, the Na_2O_2-NaOH fusion results are presented in Tables 2.2a through 2.2c, and the drainable liquid HNO_3-HCl digestion results are presented in Tables 2.3a and 2.3b. In general, the agreement between the results from the two fusion methods is good, considering the very small sample sizes used (i.e., approximately 0.2 grams) and the difficulty in obtaining homogeneous samples. An estimate of the sample detection limit can be obtained from the tables by multiplying the estimated instrument detection limit (IDL) for each analyte by the appropriate sample processing/dilution factor (Dil Fctr). It should be noted that the processing blank has not been subtracted from any of the reported sample results. Based on the crucible material used for each fusion, nickel and potassium results should be obtained from the Na_2O_2-NaOH fusions and the sodium, zirconium and phosphorus results should be obtained from the KOH-KNO_3 fusions. Of interest for the core sludge material is the significant decrease in aluminum and significant increase in calcium, iron, nickel, strontium, and uranium with depth (i.e., from Segment 1 to Segment 4).

Quality Control, Precision, and Accuracy:

The results for both drainable liquids and each segment and quarter segment sample (except Segment 2A for the Na_2O_2-NaOH fusion) are reported, along with the associated batch processing blanks. Duplicate sample results are reported for both drainable liquids and for all segments and quarter segments prepared by KOH-KNO_3 fusions; however, only Segment 1 was prepared and analyzed in duplicate from the Na_2O_2-NaOH fusion. Pre-digestion blank spike and matrix spike results are included with the drainable liquid reports, and post-fusion blank spike and matrix spike results are included with both the KOH-KNO_3 and Na_2O_2-NaOH fusion reports.

To evaluate the precision of the analytical laboratory sampling, preparation, and analysis, the relative percent difference (RPD) between duplicates is used. The RPD is shown for all samples analyzed in duplicate, and the RPD is flagged with a "*" whenever the RPD has exceeded the 10% criteria required by the TCP and the sample and duplicate results exceed 10 times the instrument detection limit (IDL). As can be seen in the tables, RPDs are very high for many of the segments and quarter segments analyzed. The inconsistency seen in the RPDs is attributed primarily to the difficulties in obtaining fully homogenized samples (either due to the limited sample available or the consistency/texture of the sample material), the complexity and diversity of each of the segments, and the very small sample sizes prepared for analysis.

The accuracy of the ICP results for the analytes of interest is estimated by either the pre/post spike recovery or percent difference (%D) from serial dilution. For serial dilution, the %D is calculated and reported when the initial sample exceeds fifty times the IDL; interferences are suspected if the reported %D exceeds 10%. Except in a very few isolated instances, the %D is well within the TCP 10% criteria. For post spikes of the initial analytical runs, the TCP requirement of 90-110% matrix spike recovery was not met for all analytes of interest. This is attributed primarily to the sample matrix or the manual dilution operation, since analytical QC samples, such as the continuing calibration verifications, have acceptable results. The $KOH-KNO_3$ and Na_2O_2-NaOH fusions post spiking reanalyses results using a special mixed spiking solution containing 31 analytes are shown in Tables 2.1e and 2.2c. Except for a few analytes (specifically, Al, Be, Na, Tl, and U on the $KOH-KNO_3$ fusions, and Al, As, Ca, Li, Sb, Tl, and U on the Na_2O_2-NaOH fusions), the TCP recovery criteria has been met; the failures are attributed to final analyte concentrations below the method detection limit (i.e., 10 times the IDL) or high analyte concentrations in the samples. In general the recoveries for the post blank spike track the recoveries for the post matrix spike, suggesting that the failures are not matrix related. Typically for those analytes demonstrating spike recoveries exceeding the 90%-110% TCP criteria, recoveries were within 85%-115%.

The processing blank provides information on contamination potentially introduced during the fusion processing. The sodium contamination in the $KOH-KNO_3$ fusion blank is above 10 times the IDL; however, since the sodium concentration is very high, the level of contamination does not impact the reported sodium results. However, iron, manganese, and boron contamination in the $KOH-KNO_3$ fusion blank render these results questionable for all but those samples containing high analyte levels. The calcium and boron contamination is sufficient enough in the Na_2O_2-NaOH fusion blank to render calcium results on Segments 1, 2A, 2D, and 3A and boron results on all segments unusable. The sodium contamination in the HNO_3-HCl acid digestion blank is above 10 times the IDL; however, the level of contamination has no effect on the reported high sodium concentration. Boron is also above 10 times the IDL, rendering the low concentration of boron found in the drainable liquid samples unreliable.

The majority of analytes reported (including the TCP analytes of interest defined in the introduction) are supported by the required verification QC (e.g., verification standards and blanks analyzed every 10 samples). However, some reported analytes (specifically, Ce, Eu, La, Nd, P, Pd, Rh, Sn, Te, Th, Te, Th, Tl, U, W and Y) have only beginning and closing QC verification, and are presented for information only. In general the analytical results for these analytes are expected to be good; however, the QC performed with the analytical run does not conform to the governing QA plan.

Additional Analyses:

Besides the analyses required by the TCP, core homogenization tests of Segment 1 and Segment 4B and water leach analyses of sludge segments, quarter segments, and drainable liquids were performed. Although no spiking was performed for these analyses, all other processing QC (e.g., duplicates and blanks) and analytical QC (e.g., verification standards) analyses were performed. The reports and data from the ICP analysis for the homogenization test samples and water leach samples can be found in Appendix C.

Table 2-1a: Tank BY-108, Core 99 Sludge, Segments 1 & 2D, ICP (KOH-KNO_3 Fusions)

Project: TWRS ICP ANALYSIS REPORT -- KOH/Ni Fusions m091395a
Procedure: PNL-ALO-211 09/13/95

Core: 99

Est. IDL ug/mL	Analyte	Sample ug/g	Dilution ug/g	%D	Duplicate ug/g	RPD	Sample ug/g	Duplicate ug/g	RPD	Batch Blk ug/g	Net PSpk ug/mL	Post Spk Rec
		Seg 1			Seg 1		Seg 2D	Seg 2D			Seg 1	
	Sam Log#:	95-07941-			95-07941-		95-07943-	95-07943-		95-07941-	95-07941-	
	Split#:	H1T-Smp			H2T-Dup		H1-Smp	H2-Dup		HCB-Blk	H1T-SmpPS	
	ICP Dil:	2.00	10.00		2.00		2.00	2.00		2.00	5.00	
	Dil Fctr:	1990.0	9950.2		1949.3		594.9	612.9		924.2	4975.1	
0.015	Ag	ND	ND		ND		ND	ND		ND	ND	
0.060	Al	135,670	141,075	4	131,644	3	70,942	67,093	6	(427)	8.45	84.5%
0.080	As	(282)	ND		(222)		(83)	(73)		ND	ND	
0.020	B	(383)	(455)		(363)		(63)	(64)		(163)	0.00	
0.010	Ba	(24)	ND		ND		ND	ND		ND	ND	
0.005	Be	ND	ND		ND		ND	ND		ND	ND	
0.100	Bi	(318)	ND		ND		ND	ND		ND	ND	
0.050	Ca	(739)	(647)		(437)		855	888	4	(80)	4.61	92.2%
0.015	Cd	ND	ND		ND		ND	ND		ND	0.24	96.3%
0.100	Ce	ND	ND		ND		ND	ND		ND	ND	
0.010	Co	(58)	(115)		(45)		(27)	(31)		(20)	(0.00)	
0.020	Cr	(146)	ND		(152)		177	173	3	ND	1.41	93.9%
0.010	Cu	ND	ND		ND		234	(23)		ND	ND	
0.050	Dy	ND	ND		ND		ND	ND		ND	ND	
0.100	Eu	ND	ND		ND		ND	ND		ND	ND	
0.020	Fe	1,488	(1,641)		971	42 *	710	1,145	47 *	270	(0.00)	
2.000	K	n/a	n/a		n/a		n/a	n/a		n/a	n/a	
0.050	La	ND	ND		ND		ND	ND		ND	ND	
0.030	Li	(109)	ND		(121)		(54)	(49)		(51)	ND	
0.100	Mg	(368)	ND		(247)		(75)	ND		ND	(0.11)	
0.005	Mn	305	(329)		269	12 *	206	285	32 *	86	0.09	92.2%
0.030	Mo	ND	ND		ND		ND	ND		ND	ND	
0.080	Na	95,300	101,561	7	95,728	0	139,877	146,959	5	1,890	13.35	
0.050	Nd	(101)	ND		ND		ND	ND		(50)	(0.00)	
0.030	Ni	n/a	n/a		n/a		n/a	n/a		n/a	n/a	
0.100	P	23,897	24,574	3	29,552	21 *	2,505	2,230	12 *	ND	(0.04)	
0.060	Pb	(351)	(704)		(256)		(106)	(94)		(86)	4.89	97.8%
0.300	Pd	ND	ND		ND		ND	ND		ND	ND	
0.300	Rh	ND	ND		ND		ND	ND		ND	ND	
0.050	Sb	(129)	ND		(106)		ND	ND		(48)	ND	
0.100	Se	ND	ND		ND		ND	ND		ND	ND	
0.500	Si	(6,917)	(7,371)		(4,939)		(1,183)	(1,150)		(837)	9.38	93.8%
1.000	Sn	ND	ND		ND		ND	ND		ND	ND	
0.005	Sr	(27)	ND		(22)		(15)	(13)		ND	0.00	
0.500	Te	ND	ND		ND		ND	ND		ND	ND	
0.800	Th	ND	ND		ND		ND	ND		ND	ND	
0.005	Ti	358	(400)		181	65 *	(23)	(22)		101	(0.00)	
0.500	Tl	ND	ND		ND		ND	ND		ND	ND	
2.000	U	ND	ND		ND		ND	ND		ND	(18.70)	93.5%
0.010	V	(57)	(105)		(48)		(7)	ND		ND	(0.00)	
0.500	W	ND	ND		ND		ND	ND		ND	ND	
0.010	Y	ND	ND		ND		ND	ND		ND	ND	
0.020	Zn	ND	ND		ND		(50)	(49)		ND	ND	
0.010	Zr	(174)	(218)		(187)		ND	ND		(62)	0.46	92.0%

Note:
1) Method Detecion Limit (MDL) = 10x IDL; "( )" results <MDL but =>IDL.
2) Above 5 times MDL, results reportable to 2 1/2 significant digits.
3) Blank is reported in ug/g "equivalence" to indicate blank effect on sample results.
4) The process "blank" has not been subtracted from the "Sample & Duplicate" results.
5) Above 5 times the MDL, precision is estimated at +/-10% and accuracy at +/-15%.
6) "ND" = Not Detected; Estimated Sample Detection Limit (ug/g) = (IDL in ug/mL) * (Dil Fctr).
7) If RPD flagged with "*", then RPD >20% and sample & duplicate results >MDL.
8) If Percent Difference (%D) >10% and sample results >5x MDL, chemical/physical interferences may exist.
9) Data, including calibration/QC, archived File ICP-325-405-1/95D256a

Table 2-1b: Tank BY-108, Core 99 Sludge, Segments 2A & 3A, ICP ($KOH-KNO_3$ Fusion)

Project: TWRS ICP ANALYSIS REPORT -- KOH/Ni Fusions m091395b
Procedure: PNL-ALO-211 09/13/95

Core 99

Est. IDL ug/mL	Analyte	Seg 2A Sam Log#: 95-07942- Split#: H1-Smp ICP Dil: 2.00 Dil Fctr: 1736.1 Sample ug/g	ICP Dil: 10.00 Dil Fctr: 8680.6 Dilution ug/g	%D	Seg 2A 95-07942- H2-Dup 2.00 1803.4 Duplicate ug/g	RPD	Seg 3A 95-07944- H1-Smp 2.00 1650.2 Sample ug/g	Seg 3A 95-07944- H2-Dup 2.00 1408.5 Duplicate ug/g	RPD	95-07942- H3-Blk 2.00 1635.0 Batch Blk ug/g
0.015	Ag	ND	ND		ND		ND	ND		(25)
0.060	Al	8,327	8,892	7	10,449	23 *	37,342	32,120	15 *	(316)
0.080	As	ND	ND		ND		ND	ND		ND
0.020	B	598	(666)		(149)		1,088	415	89 *	365
0.010	Ba	ND	ND		ND		ND	(16)		ND
0.005	Be	ND	ND		ND		ND	ND		ND
0.100	Bi	ND	ND		ND		ND	ND		ND
0.050	Ca	1,925	ND		2,164	12 *	1,132	1,035	9	(224)
0.015	Cd	ND	ND		ND		ND	ND		ND
0.100	Ce	ND	ND		ND		ND	ND		ND
0.010	Co	(44)	ND		(42)		(63)	(50)		(88)
0.020	Cr	(72)	ND		(122)		(164)	(178)		(35)
0.010	Cu	ND	ND		327		(100)	(75)		(23)
0.050	Dy	ND	ND		ND		ND	ND		ND
0.100	Eu	ND	ND		ND		ND	ND		ND
0.020	Fe	5,112	5,464	7	2,510	68 *	1,608	1,769	10	815
2.000	K	n/a	n/a		n/a		n/a	n/a		n/a
0.050	La	ND	ND		ND		ND	ND		ND
0.030	Li	ND	ND		ND		ND	ND		ND
0.100	Mg	ND	ND		ND		(199)	(147)		ND
0.005	Mn	335	(356)		298	12 *	746	535	33 *	677
0.030	Mo	ND	ND		ND		ND	ND		ND
0.080	Na	201,659	220,212	9	201,554	0	169,524	161,983	7	4,892
0.050	Nd	(108)	ND		ND		(97)	(82)		(120)
0.030	Ni	n/a	n/a		n/a		n/a	n/a		n/a
0.100	P	(709)	ND		(628)		5,405	7,079	27 *	ND
0.060	Pb	(159)	ND		(144)		(167)	(150)		(158)
0.300	Pd	ND	ND		ND		ND	ND		ND
0.300	Rh	ND	ND		ND		ND	ND		ND
0.050	Sb	(91)	ND		ND		ND	ND		(100)
0.100	Se	ND	ND		ND		ND	ND		ND
0.500	Si	ND	ND		(1,072)		(1,935)	(1,582)		ND
1.000	Sn	ND	ND		ND		ND	ND		ND
0.005	Sr	ND	ND		ND		162	201	22 *	ND
0.500	Te	ND	ND		ND		ND	ND		ND
0.800	Th	ND	ND		ND		ND	ND		ND
0.005	Ti	(22)	ND		(26)		(38)	(28)		(24)
0.500	Tl	ND	ND		ND		ND	ND		ND
2.000	U	ND	ND		ND		ND	ND		ND
0.010	V	ND	ND		ND		(22)	(22)		(18)
0.500	W	ND	ND		ND		ND	ND		ND
0.010	Y	ND	ND		ND		ND	ND		ND
0.020	Zn	(71)	ND		(250)		(103)	(98)		(75)
0.010	Zr	ND	ND		ND		ND	ND		ND

Note:
1) Method Detecion Limit (MDL) = 10x IDL; "( )" results <MDL but =>IDL.
2) Above 5 times MDL, results reportable to 2 1/2 significant digits.
3) Blank is reported in ug/g "equivalence" to indicate blank effect on sample results.
4) The process "blank" has not been subtracted from the "Sample & Duplicate" results.
5) Above 5 times the MDL, precision is estimated at +/-10% and accuracy at +/-15%.
6) "ND" = Not Detected; Estimated Sample Detection Limit (ug/g) = (IDL in ug/mL) * (Dil Fctr).
7) If RPD flagged with "*", then RPD >20% and sample & duplicate results >MDL.
8) If Percent Difference (%D) >10% and sample >5x MDL, chemical/physical interferences may exist.
9) Data, including calibration/QC, archived File ICP-325-405-1/95D256a

Table 2-1c: Tank BY-108, Core 99 Sludge, Segments 4A & 4C, ICP (KOH-KNO_3 Fusion)

Project: TWRS
Procedure: PNL-ALO-211

ICP ANALYSIS REPORT -- KOH/Ni Fusions

m091395c
09/13/95

Core 99

		Seg 4A			Seg 4A		Seg 4C	Seg 4C			Seg 4A	
	Sam Log#:	95-07945-			95-07945-		95-07947-	95-07947-		95-07945-	95-07945-	
	Split#:	H1-Smp			H2-Dup		H1-Smp	H2-Dup		H3-Blk	H1-SmpPS	
	ICP Dil:	2.00	10.00		2.00		2.00	2.00		2.00	5.00	
	Dil Fctr:	1857.0	9285.1		1706.5		1836.5	1384.1		1672.6	4642.5	
Est. IDL ug/mL	Analyte	Sample ug/g	Dilution ug/g	%D	Duplicate ug/g	RPD	Sample ug/g	Duplicate ug/g	RPD	Batch Blk ug/g	Net PSpk ug/mL	Post Spk Rec
0.015	Ag	(32)	ND		(33)		(39)	(31)		ND	ND	
0.060	Al	14,622	15,819	8	14,338	2	7,155	7,158	0	(354)	9.05	90.5%
0.080	As	ND	ND		ND		ND	(115)		ND	ND	
0.020	B	(293)	(354)		390		828	543	42 *	1,043	0.00	
0.010	Ba	257	(291)		262	2	(87)	(90)		ND	0.00	
0.005	Be	ND	ND		ND		ND	ND		ND	ND	
0.100	Bi	(1,279)	(1,350)		(1,369)		(878)	(926)		ND	(0.01)	
0.050	Ca	11,276	11,872	5	11,406	1	15,618	15,516	1	(149)	4.57	91.4%
0.015	Cd	ND	ND		ND		ND	ND		ND	0.25	98.5%
0.100	Ce	ND	ND		(214)		(202)	(188)		ND	ND	
0.010	Co	(81)	ND		(71)		(96)	(74)		(55)	(0.00)	
0.020	Cr	613	(661)		545	12 *	(90)	(79)		ND	1.41	94.3%
0.010	Cu	(128)	(157)		ND		435	269	47 *	(150)	0.01	
0.050	Dy	ND	ND		ND		ND	ND		ND	ND	
0.100	Eu	ND	ND		ND		ND	ND		ND	ND	
0.020	Fe	19,490	21,229	9	19,044	2	32,264	35,089	8	738	(0.13)	
2.000	K	n/a	n/a		n/a		n/a	n/a		n/a	n/a	
0.050	La	ND	ND		ND		ND	ND		ND	ND	
0.030	Li	ND	ND		ND		ND	ND		ND	ND	
0.100	Mg	(1,293)	(1,477)		(1,143)		(1,141)	(1,188)		ND	(0.09)	
0.005	Mn	1,024	1,105	8	467	75 *	1,069	965	10	364	0.09	88.8%
0.030	Mo	ND	ND		ND		ND	ND		ND	ND	
0.080	Na	135,458	154,142	14	121,043	11 *	111,761	114,902	3	4,494	13.25	
0.050	Nd	(143)	ND		(166)		(191)	(174)		(111)	ND	
0.030	Ni	n/a	n/a		n/a		n/a	n/a		n/a	n/a	
0.100	P	14,255	15,057	6	12,926	10	24,626	24,629	0	ND	(0.07)	
0.060	Pb	(1,075)	(1,212)		1,156		(1,047)	1,125		(152)	4.92	98.4%
0.300	Pd	ND	ND		ND		ND	ND		ND	ND	
0.300	Rh	ND	ND		ND		ND	ND		ND	ND	
0.050	Sb	ND	ND		(106)		(107)	(84)		ND	ND	
0.100	Se	ND	ND		ND		ND	(152)		ND	ND	
0.500	Si	(2,929)	ND		(4,696)		(2,175)	(2,045)		ND	9.66	96.6%
1.000	Sn	ND	ND		ND		ND	ND		ND	ND	
0.005	Sr	12,468	13,734	10	12,751	2	1,084	1,097	1	ND	(0.09)	
0.500	Te	ND	ND		ND		ND	ND		ND	ND	
0.800	Th	ND	ND		ND		ND	ND		ND	ND	
0.005	Ti	(64)	(73)		(56)		(58)	(56)		(22)	(0.00)	
0.500	Tl	ND	ND		ND		ND	ND		ND	ND	
2.000	U	(30,957)	(34,843)		(31,604)		40,875	44,374	8	ND	18.37	91.8%
0.010	V	(23)	ND		(27)		(28)	(25)		ND	ND	
0.500	W	ND	ND		ND		ND	ND		ND	ND	
0.010	Y	ND	ND		ND		(21)	(21)		ND	ND	
0.020	Zn	(297)	(305)		(228)		(354)	310		(129)	0.01	
0.010	Zr	ND	ND		ND		ND	ND		ND	0.46	91.9%

Note:
1) Method Detecion Limit (MDL) = 10x IDL; "( )" results <MDL but =>IDL.
2) Above 5 times MDL, results reportable to 2 1/2 significant digits.
3) Blank is reported in ug/g "equivalence" to indicate blank effect on sample results.
4) The process "blank" has not been subtracted from the "Sample & Duplicate" results.
5) Above 5 times the MDL, precision is estimated at +/-10% and accuracy at +/-15%.
6) "ND" = Not Detected; Estimated Sample Detection Limit (ug/g) = (IDL in ug/mL) * (Dil Fctr).
7) If RPD flagged with "*", then RPD >20% and sample & duplicate results >MDL.
8) If Percent Difference (%D) >10% and sample results >5x MDL, chemical/physical interferences may exist.
9) Data, including calibration/QC, archived File ICP-325-405-1/95D256a

Table 2-1d: Tank BY-108, Core 99 Sludge, Segments 4B & 4D, ICP (KOH-KNO_3 Fusions)

Project: TWRS
Procedure: PNL-ALO-211

ICP ANALYSIS REPORT -- KOH/Ni Fusions

m091395d
09/13/95

Core 99

		Seg 4B			Seg 4B		Seg 4D	Seg 4D		
	Sam Log#:	95-07946-			95-07946-		95-07948-	95-07948-		95-07946-
	Split#:	H1T-Smp			H2T-Dup		H1-Smp	H2-Dup		HCB-Blk
	ICP Dil:	2.00	10.00		2.00		2.00	2.00		2.00
	Dil Fctr:	1996.0	9980.0		2016.1		2024.3	2283.1		2073.6
Est. IDL ug/mL	Analyte	Sample ug/g	Dilution ug/g	%D	Duplicate ug/g	RPD	Sample ug/g	Duplicate ug/g	RPD	Batch Blk ug/g
0.015	Ag	ND	ND		(32)		(33)	(38)		(42)
0.060	Al	13,068	13,685	5	10,380	23 *	8,608	8,725	1	(1,116)
0.080	As	(220)	ND		(214)		ND	ND		(222)
0.020	B	460	(532)		405	13 *	1,119	(456)		(385)
0.010	Ba	619	(666)		445	33 *	(117)	(120)		ND
0.005	Be	ND	ND		ND		ND	ND		ND
0.100	Bi	3,245	(3,512)		2,502	26 *	(1,123)	(1,174)		(255)
0.050	Ca	11,867	12,099	2	8,471	33 *	7,973	8,038	1	(260)
0.015	Cd	(57)	ND		(76)		ND	ND		ND
0.100	Ce	ND	ND		ND		ND	ND		(255)
0.010	Co	(93)	(140)		(89)		(94)	(129)		(91)
0.020	Cr	567	(578)		(398)		(103)	(119)		(77)
0.010	Cu	(63)	(122)		(45)		247	(169)		(66)
0.050	Dy	ND	ND		ND		ND	ND		ND
0.100	Eu	ND	ND		ND		ND	ND		ND
0.020	Fe	29,045	30,298	4	21,333	31 *	44,493	44,702	0	(397)
2.000	K	n/a	n/a		n/a		n/a	n/a		n/a
0.050	La	ND	ND		ND		ND	ND		ND
0.030	Li	(127)	ND		(171)		ND	ND		ND
0.100	Mg	(1,511)	(1,798)		(1,199)		(1,233)	(1,209)		(224)
0.005	Mn	803	836	4	644	22 *	1,234	1,554	23 *	390
0.030	Mo	ND	ND		ND		ND	ND		ND
0.060	Na	103,437	113,594	10	114,857	10	117,626	117,166	0	4,504
0.050	Nd	(154)	ND		(176)		(171)	(200)		(238)
0.030	Ni	n/a	n/a		n/a		n/a	n/a		n/a
0.100	P	20,096	20,254	1	28,137	33 *	26,870	27,825	3	(364)
0.060	Pb	(1,141)	(1,454)		(902)		1,544	1,628	5	(332)
0.300	Pd	ND	ND		ND		ND	ND		(926)
0.300	Rh	ND	ND		ND		ND	ND		ND
0.050	Sb	(135)	ND		(148)		(102)	ND		(186)
0.100	Se	ND	ND		ND		ND	ND		ND
0.500	Si	(4,194)	ND		(4,532)		(2,567)	(2,746)		ND
1.000	Sn	ND	ND		ND		ND	ND		ND
0.005	Sr	27,379	28,536	4	18,725	38 *	1,452	1,471	1	(18)
0.500	Te	ND	ND		ND		ND	ND		ND
0.800	Th	ND	ND		ND		ND	ND		ND
0.005	Ti	478	513		412	15 *	(76)	(63)		322
0.500	Tl	ND	ND		ND		ND	ND		ND
2.000	U	61,811	(69,317)		46,561	28 *	51,491	51,633	0	ND
0.010	V	(26)	ND		(29)		(26)	(32)		(35)
0.500	W	ND	ND		ND		ND	ND		ND
0.010	Y	ND	ND		ND		(24)	(25)		ND
0.020	Zn	(63)	ND		ND		(396)	(368)		ND
0.010	Zr	(33)	ND		ND		ND	ND		(32)

Note:
1) Method Detecion Limit (MDL) = 10x IDL; "( )" results <MDL but =>IDL.
2) Above 5 times MDL, results reportable to 2 1/2 significant digits.
3) Blank is reported in ug/g "equivalence" to indicate blank effect on sample results.
4) The process "blank" has not been subtracted from the "Sample & Duplicate" results.
5) Above 5 times the MDL, precision is estimated at +/-10% and accuracy at +/-15%.
6) "ND" = Not Detected; Estimated Sample Detection Limit (ug/g) = (IDL in ug/mL) * (Dil Fctr).
7) If RPD flagged with "*", then RPD >20% and sample & duplicate results >MDL.
8) If Percent Difference (%D) >10% and sample results >5x MDL, chemical/physical interferences may exist.
9) Data, including calibration/QC, archived File ICP-325-405-1/95D256a

Table 2-1e: Tank BY-108, Core 99 Sludge, Post Spike Reanalysis, ICP (KOH-KNO_3 Fusion)

Project: TWRS
Procedure: PNL-ALO-211

ICP ANALYSIS REPORT -- KOH/Ni Fusions

Core 99
Post Spike Reruns (Special)

File: m091995b
Analyzed: 09/19/95

	Seg 1	Seg 1	Blank	Blank Spk
Sam Log#:	95-07941-	95-07941-	95-07941-	95-07941-
Split#:	H1T-Smp	H1T-Smp-PS	HCB-Smp	HCB-Smp-PS
ICP Dil:	5.00	5.00	5.00	5.00
Dil Fctr:	4975.1	4975.1	4975.1	4975.1
Run Time:	13:46	13:50	13:39	13:43

Est. IDL ug/mL	Post Spk Conc ug/mL	Analyte	Sample ug/g	Net PSpk ug/mL	Post Spk Recovery	Sample ug/g	Net PSpk ug/mL	Post Spk Recovery
0.015	0.50	Ag	ND	(0.62)	123.0%	ND	(0.60)	120.3%
0.060	75.00	Al	144,823	68.17	90.9%	(1,119)	69.05	92.1%
0.080	2.50	As	ND	(2.83)	113.2%	ND	(2.69)	107.7%
0.020	2.00	B	(442)	1.87	93.7%	(405)	1.84	92.0%
0.010	0.50	Ba	ND	(0.50)	99.7%	ND	(0.47)	94.3%
0.005	0.10	Be	ND	(0.09)	92.1%	ND	(0.09)	89.3%
0.100	25.00	Bi	ND	24.11	96.4%	ND	23.23	92.9%
0.050	50.00	Ca	(749)	48.43	96.9%	(363)	46.80	93.6%
0.015	1.75	Cd	ND	1.74	99.5%	ND	1.73	98.7%
0.100		Ce	ND	ND		ND	ND	
0.010	1.00	Co	(66)	1.00	100.1%	(55)	0.98	98.4%
0.020	8.50	Cr	(139)	8.37	98.4%	ND	8.18	96.3%
0.010	1.00	Cu	ND	0.98	97.9%	ND	0.92	92.3%
0.050		Dy	ND	ND		ND	ND	
0.100		Eu	ND	ND		ND	ND	
0.020	20.00	Fe	1,606	19.61	98.1%	(674)	19.13	95.7%
2.000	125.00	K	n/a	n/a	n/a	n/a	n/a	n/a
0.050		La	ND	ND		ND	ND	
0.030	2.00	Li	ND	1.87	93.3%	ND	1.82	91.1%
0.100	25.00	Mg	(571)	26.81	107.3%	ND	26.53	106.1%
0.005	1.00	Mn	329	0.98	98.1%	(204)	0.96	95.6%
0.030	1.00	Mo	ND	(1.01)	101.1%	ND	(1.01)	101.1%
0.080	106.85	Na	107,789	94.87	88.8%	4,976	95.32	89.2%
0.050		Nd	ND	ND		ND	(0.27)	
0.030	26.00	Ni	n/a	n/a	n/a	n/a	n/a	n/a
0.100		P	24,798	(0.29)		ND	ND	
0.060	27.50	Pb	(448)	28.33	103.0%	(346)	27.68	100.7%
0.300		Pd	ND	ND		ND	ND	
0.300		Rh	ND	ND		ND	ND	
0.050	2.50	Sb	(259)	2.66	106.4%	ND	2.73	109.4%
0.100	2.50	Se	ND	(2.58)	103.1%	ND	(2.57)	102.7%
0.500	70.00	Si	(7,343)	66.81	95.4%	ND	67.10	95.9%
1.000		Sn	ND	ND		ND	ND	
0.005	0.50	Sr	(31)	0.48	95.3%	ND	0.47	94.2%
0.500		Te	ND	ND		ND	ND	
0.800		Th	ND	ND		ND	ND	
0.005	1.00	Ti	390	0.96	95.6%	(246)	0.94	93.7%
0.500	2.50	Tl	ND	(2.93)	117.0%	ND	(2.61)	104.3%
2.000	100.00	U	ND	(91.13)	91.1%	ND	(88.99)	89.0%
0.010	1.00	V	(69)	0.95	95.2%	ND	0.96	96.1%
0.500		W	ND	ND		ND	ND	
0.010		Y	ND	ND		ND	ND	
0.020	1.00	Zn	(109)	1.00	100.2%	ND	1.03	103.2%
0.010	4.50	Zr	(185)	4.31	95.8%	(150)	4.18	92.8%

Note: 1) Method Detecion Limit (MDL) = 10x IDL; "( )" results <MDL but =>IDL.
2) Above 5 times MDL, results reportable to 2 1/2 significant digits.
3) Blank is reported in ug/g "equivalence" to indicate blank effect on sample results.
4) The process "blank" has not been subtracted from the "Sample & Duplicate" results.
5) Above 5 times the MDL, precision is estimated at +/-10% and accuracy at +/-15%.
6) "ND" = Not Detected; Estimated Sample Detection Limit (ug/g) = (IDL in ug/mL) * (Dil Fctr).

7) Data, including calibration/QC, archived File ICP-325-405-1/95D262a

Table 2-2a: Tank BY-108, Core 99 Sludge, Segments 1, 2D, & 3A, ICP (Na_2O_2-NaOH Fusion)

Project: TWRS
Procedure: PNL-ALO-211

ICP ANALYSIS REPORT -- Na2O2/Zr Fusions

Core 99

File: m091295c
Analyzed: 09/12/95

Est. IDL ug/mL	Analyte	Sample ug/g	Dilution ug/g	%D	Duplicate ug/g	RPD	Sample ug/g	Sample ug/g	Batch Blk ug/g	Net PSpk ug/mL	Post Spk Recovery
		Seg 1			Seg 1		Seg 2D	Seg 3A		Seg 1	
	Sam Log#:	95-07941-			95-07941-		95-07943-	95-07944-	95-07941-	95-07941-	
	Split#:	N1-Smp			N2-Dup		N1-Smp	N1-Smp	N3-Blk	N1-Smp-PS	
	ICP Dil:	2.00	10.00		2.00		2.00	2.00	2.00	5.00	
	Dil Fctr:	1511.7	7558.6		2114.2		886.5	1506.0	1366.8	3779.3	
	Run Time:	15:39	15:35		15:59		16:10	17:10	15:30	15:45	
0.015	Ag	ND	ND		ND		ND	ND	ND	(0.08)	
0.060	Al	104,424	115,669	11	135,410	26 *	60,889	25,702	(123)	33.00	66.0%
0.080	As	(167)	ND		(256)		(112)	ND	ND	(0.22)	
0.020	B	694	(779)		1,038	40 *	(147)	635	417	0.03	
0.010	Ba	(15)	ND		(23)		(13)	(17)	ND	ND	
0.005	Be	ND	ND		ND		ND	ND	ND	ND	
0.100	Bi	ND	ND		ND		ND	ND	ND	ND	
0.050	Ca	3,071	(3,150)		3,978	26 *	1,759	3,257	2,665	20.45	81.8%
0.015	Cd	ND	ND		ND		ND	ND	ND	1.09	87.5%
0.100	Ce	ND	ND		ND		ND	ND	ND	(0.59)	
0.010	Co	(20)	ND		(35)		(19)	(22)	ND	(0.05)	
0.020	Cr	(127)	ND		(169)		(176)	(154)	(35)	6.30	84.0%
0.010	Cu	(90)	(83)		(43)		(20)	(32)	(16)	(0.00)	
0.050	Dy	ND	ND		ND		ND	ND	ND	ND	
0.100	Eu	ND	ND		ND		ND	ND	ND	ND	
0.020	Fe	1,028	(1,190)		1,411	31 *	544	1,247	(195)	0.03	
2.000	K	(4,498)	ND		(8,232)		(4,416)	(3,879)	ND	(87.97)	88.0%
0.050	La	ND	ND		ND		ND	ND	ND	ND	
0.030	Li	(52)	ND		(60)		(60)	(48)	ND	ND	
0.100	Mg	(186)	ND		(313)		(126)	ND	ND	(0.79)	
0.005	Mn	(24)	ND		(29)		(13)	(15)	ND	0.42	83.7%
0.030	Mo	ND	ND		ND		ND	ND	ND	ND	
0.080	Na	n/a	n/a		n/a		n/a	n/a	n/a	n/a	
0.050	Nd	(125)	ND		(232)		(108)	(127)	(88)	(0.32)	
0.030	Ni	473	(530)		674	35 *	532	836	(314)	21.52	86.1%
0.100	P	3,821	(4,259)		(715)		(233)	(490)	ND	0.04	
0.060	Pb	(232)	ND		(345)		(177)	(177)	(85)	22.63	90.5%
0.300	Pd	ND	ND		ND		ND	ND	ND	ND	
0.300	Rh	ND	ND		ND		ND	ND	ND	ND	
0.050	Sb	ND	ND		(111)		(58)	ND	ND	ND	
0.100	Se	ND	ND		ND		ND	ND	ND	ND	
0.500	Si	(2,650)	ND		(4,150)		(1,127)	(1,086)	ND	41.99	84.0%
1.000	Sn	ND	ND		ND		ND	ND	ND	ND	
0.005	Sr	(58)	(67)		(70)		(35)	310	(41)	(0.00)	
0.500	Te	ND	ND		ND		ND	ND	ND	ND	
0.800	Th	ND	ND		ND		ND	ND	ND	ND	
0.005	Ti	(45)	(56)		(70)		(22)	(20)	(10)	(0.03)	
0.500	Tl	ND	ND		ND		ND	ND	ND	ND	
2.000	U	ND	ND		ND		ND	ND	ND	(85.86)	85.9%
0.010	V	(57)	(79)		(55)		(15)	(19)	ND	(0.05)	
0.500	W	ND	ND		ND		ND	ND	ND	ND	
0.010	Y	ND	ND		ND		ND	ND	ND	ND	
0.020	Zn	(42)	ND		(50)		(32)	(69)	ND	ND	
0.010	Zr	n/a	n/a		n/a		n/a	n/a	n/a	n/a	n/a

Note:
1) Method Detecion Limit (MDL) = 10x IDL; "( )" results <MDL but =>IDL.
2) Above 5 times MDL, results reportable to 2 1/2 significant digits.
3) Blank is reported in ug/g "equivalence" to indicate blank effect on sample results.
4) The process "blank" has not been subtracted from the "Sample & Duplicate" results.
5) Above 5 times the MDL, precision is estimated at +/-10% and accuracy at +/-15%.
6) "ND" = Not Detected; Estimated Sample Detection Limit (ug/g) = (IDL in ug/mL) * (Dil Fctr).
7) If RPD flagged with "*", then RPD >20% and sample & duplicate results >MDL.
8) If Percent Difference (%D) >10% and sample results >5x MDL, chemical/physical interferences may exist.
9) Data, including calibration/QC, archived File ICP-325-405-1/95D255a

Table 2-2b: Tank BY-108, Core 99 Sludge, Segments 4A, 4B, 4C, & 4D, ICP (Na_2O_2-NaOH Fusion)

Project: TWRS
Procedure: PNL-ALO-211

ICP ANALYSIS REPORT -- Na2O2/Zr Fusions

File: m091295d
Analyzed: 09/12/95

Core 99

		Seg 4A			Seg 4B	Seg 4C	Seg 4D		Seg 4A	
	Sam Log#:	95-07945-			95-07946-	95-07947-	95-07948-	95-07941-	95-07945-	
	Split#:	N1-Smp			N1-Smp	N1-Smp	N1-Smp	N3-Blk	N1-Smp-PS	
	ICP Dil:	2.00	10.00		2.00	2.00	2.00	2.00	5.00	
	Dil Fctr:	1926.8	9633.9		1857.0	1912.0	1816.5	1877.1	4817.0	
	Run Time:	17:19	17:15		17:30	17:39	18:02	15:30	17:22	
Est. IDL ug/mL	Analyte	Sample ug/g	Dilution ug/g	%D	Sample ug/g	Sample ug/g	Sample ug/g	Batch Blk ug/g	Net PSpk ug/mL	Post Spk Recovery
0.015	Ag	ND	ND		ND	ND	ND	ND	ND	
0.060	Al	15,478	16,543	7	11,193	7,430	8,166	(169)	45.50	91.0%
0.080	As	ND	ND		ND	ND	ND	ND	ND	
0.020	B	404	(475)		433	(254)	397	572	(0.03)	
0.010	Ba	265	(321)		556	(99)	(115)	ND	(0.02)	
0.005	Be	ND	ND		ND	ND	ND	ND	ND	
0.100	Bi	(1,378)	(1,428)		2,823	(933)	(1,075)	ND	(0.10)	
0.050	Ca	16,028	16,885	5	15,459	20,636	11,455	3,660	23.71	94.8%
0.015	Cd	ND	ND		ND	ND	ND	ND	1.20	96.2%
0.100	Ce	ND	ND		ND	ND	ND	ND	ND	
0.010	Co	(41)	ND		(38)	(34)	(37)	ND	(0.02)	
0.020	Cr	619	(664)		479	(97)	(109)	(48)	7.22	96.2%
0.010	Cu	(36)	ND		(31)	(66)	(105)	(22)	ND	
0.050	Dy	ND	ND		ND	ND	ND	ND	ND	
0.100	Eu	ND	ND		ND	ND	ND	ND	ND	
0.020	Fe	20,151	22,079	10	27,212	34,605	41,981	(268)	1.15	
2.000	K	(4,826)	ND		(4,904)	(4,549)	(4,777)	ND	(87.50)	87.5%
0.050	La	ND	ND		ND	ND	ND	ND	ND	
0.030	Li	ND	ND		ND	ND	ND	ND	ND	
0.100	Mg	(1,480)	(1,664)		(1,459)	(1,280)	(1,374)	ND	(0.69)	
0.005	Mn	300	(330)		328	366	498	ND	0.49	97.8%
0.030	Mo	ND	ND		ND	ND	ND	ND	ND	
0.080	Na	n/a	n/a		n/a	n/a	n/a	n/a	n/a	
0.050	Nd	(145)	ND		(161)	(174)	(199)	(120)	ND	
0.030	Ni	14,673	16,121	10	16,741	6,710	7,209	(432)	25.37	101.5%
0.100	P	(623)	ND		3,640	(1,605)	(1,437)	ND	(0.25)	
0.060	Pb	(1,154)	(1,370)		(995)	(1,144)	2,856	(117)	25.11	100.5%
0.300	Pd	ND	ND		ND	ND	ND	ND	ND	
0.300	Rh	ND	ND		ND	ND	ND	ND	ND	
0.050	Sb	ND	ND		ND	ND	ND	ND	ND	
0.100	Se	ND	ND		ND	ND	ND	ND	ND	
0.500	Si	(2,720)	ND		(2,287)	(1,554)	(1,784)	ND	48.66	97.3%
1.000	Sn	ND	ND		ND	ND	ND	ND	ND	
0.005	Sr	13,885	15,346	11	25,031	1,260	1,468	(57)	1.04	
0.500	Te	ND	ND		ND	ND	ND	ND	ND	
0.800	Th	ND	ND		ND	ND	ND	ND	ND	
0.005	Ti	(41)	(50)		(46)	(34)	(40)	(13)	(0.01)	
0.500	Tl	ND	ND		ND	ND	ND	ND	ND	
2.000	U	(33,219)	(38,672)		54,717	46,937	50,669	ND	97.64	97.6%
0.010	V	ND	ND		(20)	ND	(21)	ND	ND	
0.500	W	ND	ND		ND	ND	ND	ND	ND	
0.010	Y	ND	ND		(20)	(20)	(25)	ND	ND	
0.020	Zn	(235)	(243)		(150)	(247)	(303)	ND	0.01	
0.010	Zr	n/a	n/a		n/a	n/a	n/a	n/a	n/a	n/a

Note:
1) Method Detecion Limit (MDL) = 10x IDL; "( )" results <MDL but =>IDL.
2) Above 5 times MDL, results reportable to 2 1/2 significant digits.
3) Blank is reported in ug/g "equivalence" to indicate blank effect on sample results.
4) The process "blank" has not been subtracted from the "Sample & Duplicate" results.
5) Above 5 times the MDL, precision is estimated at +/-10% and accuracy at +/-15%.
6) "ND" = Not Detected; Estimated Sample Detection Limit (ug/g) = (IDL in ug/mL) * (Dil Fctr).
7) If RPD flagged with "*", then RPD >20% and sample & duplicate results >MDL.
8) If Percent Difference (%D) >10% and sample results >5x MDL, chemical/physical interferences may exist.
9) Data, including calibration/QC, archived File ICP-325-405-1/95D255a

Table 2-2c: Tank BY-108, Core 99 Sludge, Post Spike Reanalysis, ICP (Na_2O_2-NaOH Fusion)

Project: TWRS
Procedure: PNL-ALO-211

ICP ANALYSIS REPORT -- Na2O2/Zr Fusions

File: m091995a
Analyzed: 09/19/95

Core 99
Post Spike Reruns (Special)

			Seg 1	Seg 1		Blank	Blank Spk	
		Sam Log#:	95-07941-	95-07941-		95-07941-	95-07941-	
		Split#:	N1-Smp	N1-Smp-PS		N3-Blk	N3-Blk-PS	
		ICP Dil:	5.00	5.00		5.00	5.00	
		Dil Fctr:	3779.3	3779.3		3779.3	3779.3	
		Run Time:	14:16	14:20		14:08	14:12	
Est. IDL ug/mL	Post Spk Conc ug/mL	Analyte	Sample ug/g	Net PSpk ug/mL	Post Spk Recovery	Sample ug/g	Net PSpk ug/mL	Post Spk Recovery
0.015	0.50	Ag	ND	(0.61)	121.1%	(64)	(0.58)	115.5%
0.060	75.00	Al	122,327	64.48	86.0%	(321)	71.44	95.3%
0.080	2.50	As	ND	(2.80)	111.8%	ND	(2.81)	112.3%
0.020	2.00	B	836	1.85	92.4%	(516)	1.90	95.1%
0.010	0.50	Ba	ND	(0.48)	96.6%	ND	(0.48)	96.4%
0.005	0.10	Be	ND	(0.09)	90.2%	ND	(0.09)	90.3%
0.100	25.00	Bi	ND	23.74	95.0%	ND	24.30	97.2%
0.050	50.00	Ca	4,222	47.04	94.1%	7,612	43.51	87.0%
0.015	1.75	Cd	ND	1.72	98.0%	ND	1.78	101.5%
0.100		Ce	ND	ND		ND	ND	
0.010	1.00	Co	ND	1.03	102.9%	ND	1.05	105.0%
0.020	8.50	Cr	(150)	8.19	96.3%	ND	8.39	98.6%
0.010	1.00	Cu	(130)	0.96	95.6%	(60)	0.98	97.9%
0.050		Dy	ND	ND		ND	ND	
0.100		Eu	ND	ND		ND	ND	
0.020	20.00	Fe	1,209	19.18	95.9%	(262)	19.53	97.7%
2.000	125.00	K	ND	122.60	98.1%	ND	125.64	100.5%
0.050		La	ND	ND		ND	ND	
0.030	2.00	Li	ND	1.79	89.3%	ND	1.77	88.7%
0.100	25.00	Mg	ND	26.85	107.4%	ND	27.31	109.2%
0.005	1.00	Mn	(30)	0.96	96.2%	ND	0.99	99.2%
0.030	1.00	Mo	ND	(1.00)	100.1%	ND	(1.01)	101.0%
0.080	106.85	Na	n/a	n/a	n/a	n/a	n/a	n/a
0.050		Nd	ND	(0.30)		(208)	(0.07)	
0.030	26.00	Ni	(575)	25.60	98.5%	(410)	25.97	99.9%
0.100		P	4,074	0.11		ND	ND	
0.060	27.50	Pb	(273)	28.01	101.8%	ND	28.78	104.7%
0.300		Pd	ND	ND		ND	ND	
0.300		Rh	ND	ND		ND	ND	
0.050	2.50	Sb	ND	2.79	111.6%	ND	2.78	111.4%
0.100	2.50	Se	ND	(2.50)	99.9%	ND	(2.62)	105.0%
0.500	70.00	Si	(3,046)	66.27	94.7%	ND	68.12	97.3%
1.000		Sn	ND	ND		ND	ND	
0.005	0.50	Sr	(69)	0.46	92.7%	(54)	0.47	93.5%
0.500		Te	ND	ND		ND	ND	
0.800		Th	ND	ND		ND	ND	
0.005	1.00	Ti	(53)	0.94	94.3%	ND	0.98	98.2%
0.500	2.50	Tl	ND	(2.88)	115.3%	ND	(2.64)	105.6%
2.000	100.00	U	ND	(88.92)	88.9%	ND	(87.75)	87.8%
0.010	1.00	V	(65)	0.94	93.5%	ND	0.98	98.1%
0.500		W	ND	ND		ND	ND	
0.010		Y	ND	ND		ND	ND	
0.020	1.00	Zn	ND	1.10	110.0%	(89)	0.97	97.1%
0.010	4.50	Zr	n/a	n/a	n/a	n/a	n/a	n/a

Note:
1) Method Detecion Limit (MDL) = 10x IDL; "( )" results <MDL but =>IDL.
2) Above 5 times MDL, results reportable to 2 1/2 significant digits.
3) Blank is reported in ug/g "equivalence" to indicate blank effect on sample results.
4) The process "blank" has not been subtracted from the "Sample & Duplicate" results.
5) Above 5 times the MDL, precision is estimated at +/-10% and accuracy at +/-15%.
6) "ND" = Not Detected; Estimated Sample Detection Limit (ug/g) = (IDL in ug/mL) * (Dil Fctr).

7) Data, including calibration/QC, archived File ICP-325-405-1/950262a

Table 2-3a: Tank BY-108, Core 99 Drainable Liquid, Segment 2, ICP (Acid Digestion)

Project: TWRS
Procedure: PNL-ALO-211
M&TE: JA ICP WB73520

ICP ANALYSIS REPORT
** Sample Results -- Acid Digestion **

Core 99 Drainable Liquids

Analyze Date: 09/12/95
File: m091295a
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Samp Log#:	Seg 2 95-07932- A1 Sam	95-07932- A1 SamDil		Seg 2 95-07932- A2 Dup	95-07932- A2 DupDil		95-07932- A3 Blank		**Estimated**	
Dil Fctr:	125.00	625.00		125.00	625.00		12.50			
ICP Dil:	10.00	50.00		10.00	50.00		1.00			
	Sample			Duplicate			Blank		IDL	MDL
Analyte	ug/mL	ug/mL	%D	ug/mL	ug/mL	%D	ug/mL		ug/mL	ug/mL
Ag	ND	ND		ND	ND		ND		0.015	0.1500
Al	35,454	36,139	2%	34,929	34,667	1%	(2)		0.060	0.6000
As	(30)	ND		(32)	ND		ND		0.080	0.8000
B	43	(43)		40	(41)		20	>MDL	0.020	0.2000
Ba	ND	ND		ND	ND		ND		0.010	0.1000
Be	ND	ND		ND	ND		ND		0.005	0.0500
Bi	ND	ND		ND	ND		ND		0.100	1.0000
Ca	ND	ND		ND	ND		ND		0.050	0.5000
Cd	ND	ND		ND	ND		ND		0.015	0.1500
Ce	ND	ND		ND	ND		ND		0.100	1.0000
Co	(9)	(8)		(9)	(9)		ND		0.010	0.1000
Cr	280	285	2%	277	277	0%	ND		0.020	0.2000
Cu	(4)	ND		(4)	ND		ND		0.010	0.1000
Dy	ND	ND		ND	ND		ND		0.050	0.5000
Eu	ND	ND		ND	ND		ND		0.100	1.0000
Fe	118	(123)		117	(120)		ND		0.020	0.2000
K	2,656	(2,716)		2,614	(2,713)		ND		2.000	20.0000
La	ND	ND		ND	ND		ND		0.050	0.5000
Li	ND	ND		ND	ND		ND		0.030	0.3000
Mg	ND	ND		ND	ND		ND		0.100	1.0000
Mn	ND	ND		ND	ND		ND		0.005	0.0500
Mo	(14)	ND		(14)	ND		ND		0.030	0.3000
Na	OvrRng	201,647		OvrRng	188,042	8%	24	>MDL	0.080	0.8000
Nd	ND	ND		ND	ND		ND		0.050	0.5000
Ni	310	319	3%	306	314	3%	ND		0.030	0.3000
P	467	(456)		462	(453)		ND		0.100	1.0000
Pb	91	(76)		91	(86)		ND		0.060	0.6000
Pd	ND	ND		ND	ND		ND		0.300	3.0000
Rh	ND	ND		ND	ND		ND		0.300	3.0000
Sb	ND	ND		ND	ND		ND		0.050	0.5000
Se	ND	ND		ND	ND		ND		0.100	1.0000
Si	ND	ND		ND	ND		(25)		0.500	5.0000
Sn	ND	ND		ND	ND		ND		1.000	10.0000
Sr	ND	ND		ND	ND		ND		0.005	0.0500
Te	ND	ND		ND	ND		ND		0.500	5.0000
Th	ND	ND		ND	ND		ND		0.800	8.0000
Ti	ND	ND		ND	ND		ND		0.005	0.0500
Tl	(76)	ND		(75)	ND		ND		0.500	5.0000
U	ND	ND		ND	ND		ND		2.000	20.0000
V	ND	ND		ND	ND		ND		0.010	0.1000
W	(63)	ND		ND	ND		ND		0.500	5.0000
Y	ND	ND		ND	ND		ND		0.010	0.1000
Zn	(7)	ND		(7)	ND		ND		0.020	0.2000
Zr	ND	ND		ND	ND		ND		0.010	0.1000

Note:
1) Above 5 times MDL, results reportable to 2 1/2 significant digits. "( )" results <MDL but =>IDL.
2) Blank is reported adjusted for the an "average sample volume".
3) The process "blank" has not been subtracted from the "Sample & Duplicate" results.
4) Above 5 times the MDL, precision is estimated at +/-10% and accuracy at +/-15%.
5) "ND" = Not Detected; Estimated Sample Detection Limit (ug/mL) = (IDL in ug/mL) * (Dil Fctr).
6) If Percent Difference (%D) >10% and sample results >5X MDL, chemical/physical interferences may exist.
7) ">MDL" indicates analyte found in process blank above the MDL.

8) Data, including calibration/QC, archived File ICP-325-405-1/95D255a

Table 2-3a: **Tank BY-108, Core 99 Drainable Liquid, Segment 2, ICP (Acid Digestion) - cont.**

Project: TWRS
Procedure: PNL-ALO-211
M&TE: JA ICP WB73520

ICP ANALYSIS REPORT
**** QC Results *****

09/12/95 : Analyzed
m091295a : File
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				Samp Log# >>>		Seg 2 95-07932-A4 Smp+Spk			95-07932-A5 Blk Spk			Post Spk			
				Spk Dil Fctr >>		100.00			100.00						
				Sam Dil Fctr >>		125.00									
				ICP Dil >>>		10.00			1.00						
Analyte ↓	Average A1 & A2 ug/mL	20% RPD	Flg	PreSpk STD ug/mL	Spk Conc in Sam/Blk ug/mL	Net Spk Conc ug/mL	Rec	Spk Flg	Net Blk Spk Cntrl ug/mL	Rec	10% Spk Flg	PostSpk STD ug/mL	Post Spike ug/mL	Rec	Spk Flg
Ag	---														
Al	35,192	1%		1000	10.000	12.93	129.3%	N	8.619	86.2%	N				
As	---														
B	42	6%													
Ba	---														
Be	---														
Bi	---														
Ca	---			500	5.000	5.92	118.5%	N	4.236	84.7%					
Cd	---			25	0.250	0.24	95.4%		0.207	82.6%					
Ce	---														
Co	---														
Cr	279	1%		150	1.500	1.38	92.0%		1.314	87.6%					
Cu	---														
Dy	---														
Eu	---														
Fe	118	1%													
K	2,635	2%		2000	20.000	20.69	103.4%		16.984	84.9%					
La	---														
Li	---														
Mg	---														
Mn	---			10	0.100	0.09	94.4%		0.088	87.7%					
Mo	---														
Na	194,845	7%													
Nd	---														
Ni	308	1%		500	5.000	4.70	94.1%		4.539	90.8%					
P	465	1%													
Pb	91	1%		500	5.000	4.92	98.5%		4.373	87.5%					
Pd	---														
Rh	---														
Sb	---														
Se	---														
Si	---			1000	10.000	10.53	105.3%		9.833	98.3%					
Sn	---														
Sr	---														
Te	---														
Th	---														
Ti	---														
Tl	---														
U	---			2000	20.000	19.64	98.2%		17.555	87.8%					
V	---														
W	---														
Y	---														
Zn	---														
Zr	---			50	0.500	0.41	n/a		0.438	87.6%					

Note:
1) Above 5 times MDL, results reportable to 2 1/2 significant digits. "( )" results <MDL but =>IDL.
2) Above 5 times MDL, precision is estimated at +/-10% and accuracy at +/-15%.
3) Where "Blank" results are >IDL the blank has been subtracted from the "Spike Control".
4) Spike Flag (N) indicates spike is outside the QC recovery criteria.
5) If 20% flag = "*", Then RPD >20% and both sample results >MDL.
6) If spike is <25% of sample concentration, Rec is not calculated as indicated by the "n/a".
7) If sample or duplicate results are <MDL then average is not calculated (i.e., "---").

Table 2-3b: Tank BY-108, Core 99 Drainable Liquid, Segment 3, ICP (Acid Digestion)

Project: TWRS
Procedure: PNL-ALO-211
M&TE: JA ICP WB73520

ICP ANALYSIS REPORT
** Sample Results -- Acid Digestion **

Core 99 Drainable Liquids

Analyze Date: 09/12/95
File: m091295b
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	Seg 3			Seg 3						
Samp Log#:	95-07935-A1 Sam	95-07935-A1 SamDil		95-07935-A2 Dup	95-07935-A2 DupDil					
Dil Fctr:	125.00	625.00		125.00	625.00					
ICP Dil:	10.00	50.00		10.00	50.00				**Estimated**	
	<---------Sample--------->			<---------Duplicate--------->			20%		IDL	MDL
Analyte	ug/mL	ug/mL	%D	ug/mL	ug/mL	%D	RPD	Flg	ug/mL	ug/mL
Ag	ND	ND		ND	ND				0.015	0.1500
Al	37,441	37,802	1%	37,429	38,529	3%	0%		0.060	0.6000
As	(34)	ND		(32)	ND				0.080	0.8000
B	44	(46)		45	(49)		0%		0.020	0.2000
Ba	ND	ND		ND	ND				0.010	0.1000
Be	ND	ND		ND	ND				0.005	0.0500
Bi	ND	ND		ND	ND				0.100	1.0000
Ca	ND	ND		ND	ND				0.050	0.5000
Cd	ND	ND		ND	ND				0.015	0.1500
Ce	ND	ND		ND	ND				0.100	1.0000
Co	(9)	(9)		(9)	(9)				0.010	0.1000
Cr	294	304	3%	291	300	3%	1%		0.020	0.2000
Cu	(10)	(8)		(10)	(8)				0.010	0.1000
Dy	ND	ND		ND	ND				0.050	0.5000
Eu	ND	ND		ND	ND				0.100	1.0000
Fe	97	(101)		104	(109)		7%		0.020	0.2000
K	2,780	(2,787)		2,811	(2,836)		1%		2.000	20.0000
La	ND	ND		ND	ND				0.050	0.5000
Li	ND	ND		ND	ND				0.030	0.3000
Mg	ND	ND		ND	ND				0.100	1.0000
Mn	ND	ND		ND	ND				0.005	0.0500
Mo	(15)	ND		(15)	ND				0.030	0.3000
Na	OvrRng	185,321		OvrRng	194,961		5%		0.080	0.8000
Nd	ND	ND		ND	ND				0.050	0.5000
Ni	238	247	4%	243	256	5%	2%		0.030	0.3000
P	452	(453)		457	(454)		1%		0.100	1.0000
Pb	100	(99)		97	(88)		2%		0.060	0.6000
Pd	ND	ND		ND	ND				0.300	3.0000
Rh	ND	ND		ND	ND				0.300	3.0000
Sb	ND	ND		ND	ND				0.050	0.5000
Se	ND	ND		ND	ND				0.100	1.0000
Si	(108)	ND		(119)	ND				0.500	5.0000
Sn	ND	ND		ND	ND				1.000	10.0000
Sr	(2)	ND		(5)	(5)				0.005	0.0500
Te	ND	ND		ND	ND				0.500	5.0000
Th	ND	ND		ND	ND				0.800	8.0000
Ti	ND	ND		ND	ND				0.005	0.0500
Tl	(77)	ND		(77)	ND				0.500	5.0000
U	ND	ND		ND	ND				2.000	20.0000
V	ND	ND		ND	ND				0.010	0.1000
W	776	(780)		772	(777)		1%		0.500	5.0000
Y	ND	ND		ND	ND				0.010	0.1000
Zn	(12)	ND		(12)	ND				0.020	0.2000
Zr	ND	ND		ND	ND				0.010	0.1000

Note:
1) Above 5 times MDL, results reportable to 2 1/2 significant digits. "( )" results <MDL but =>IDL.
2) Blank is reported adjusted for the an "average sample volume".
3) The process "blank" has not been subtracted from the "Sample & Duplicate" results.
4) Above 5 times the MDL, precision is estimated at +/-10% and accuracy at +/-15%.
5) "ND" = Not Detected; Estimated Sample Detection Limit (ug/mL) = (IDL in ug/mL) * (Dil Fctr).
6) If Percent Difference (%D) >10% and sample results >5X MDL, chemical/physical interferences may exist.
7) If 20% flag = "*", then RPD >20% AND both sample and duplicate results >MDL.

8) Data, including calibration/QC, archived File ICP-325-405-1/ 950255a

Ion Chromatographic Analysis

Sample Preparation and Analysis:

The IC analyses were performed on the water leaches prepared from homogenized material from Core 99 Segment 1 and Quarter Segments 2A, 2D, 3A, and 4A through 4D and on dilutions of the drainable liquids from Segment 2 and Segment 3. The water leach samples were prepared following procedures PNL-ALO-103, "Water Leach of Sludges, Soils, and Other Solid Samples." The leaches and dilutions were performed in the SAL with sample and QC solution aliquots being transferred to the Inorganic Analysis Group for IC analysis. All IC analyses for the anions fluoride, chloride, nitrite, nitrate, phosphate, and sulfate were performed on a Dionex 4500i Ion Chromatograph system following procedure PNL-ALO-212, "Determination of Inorganic Anions by Ion Chromatography."

Analytical Results:

The IC analytical results are presented in Tables 2.4a through 2.4e. The IC analyses were conducted on 9/14/95, 9/18/95, and 9/28/95; the results reported are restricted to the 9/14/95 and 9/28/95 runs due to consistent failure of the verification standards on 9/18/95. The results for the drainable liquids are reported in μg/mL and the results for the water leaches of the sludges in μg/g. After applying the dilution and leaching processing factors, the typical detection limits are 30 μg/mL (or 30 μg/g) halides and 50 μg/mL (or 50 μg/g) oxy-anions. However, due to the necessity to dilute the samples to assure that all reported results fell within the calibration range, some reported detection limits are adjusted for additional analytical dilutions.

Quality Control, Precision, and Accuracy:

Sample and duplicate results are reported for both drainable liquids and water leaches of the sludge segment and quarter segment samples. Matrix spikes, blank spikes, and leach processing blanks are also reported for the sludge samples, as well as a dilution blank for drainable liquids. Significant difficulties were experienced with fluoride and phosphate analyses. The fluoride is impacted by significant interferences (most probably from organic anions which elute near the same retention times a fluoride), and the phosphate appeared to experience a matrix-related interference or column degradation (i.e., the verification standard for phosphate experienced consistent failure). The phosphate is considered to be reasonably reliable since ICP phosphorus analysis of the same samples confirms the reported phosphate results; however, the fluoride results are considered best available estimates. Beside phosphate and fluoride, nitrite and sulfate also slightly degrades with time and additional sample loading on the column. Based on the continuing calibration verifications standards, nitrite and sulfate may be bias high by as much as ten percent.

The RPD between duplicates is used to evaluate the precision of the sample processing, analytical sampling, and IC analysis. The majority of RPD values meet the TCP 10% criteria; however, a few anions demonstrate very poor RPDs (i.e., up to 90%) for specific samples. The poor RPDs are attributed primarily to the inconsistency of the sample material and/or the inability to obtain fully representative analytical samples. Reanalyses were performed for the majority of the samples exhibiting greater than 20% RPD; the reanalyses confirmed the original results. Very large RPD are restricted primarily to Segment 4 samples (4A through 4D); this is not unrealistic since Segment 4 is most likely the tank heel and may represent the largest variability in the tank material.

The accuracy of the IC results is estimated by the matrix spike recovery. Low recoveries on matrix spikes provide indications of matrix interferences which may adversely affect the reported analytical results. The matrix spiking solution was prepared based on the best available information on the anion concentrations suspected within Tank BY-108. Unfortunately, many of the matrix spikes are unusable due to the fact that the tank concentrations are significantly higher than anticipated. The spikes for Cl, NO_2, and NO_3 for the drainable liquids, F and PO_4 for the sludges could not be recovered due to the high concentration of the anions in the samples. The <u>only</u> matrix spike

spike meeting the 90-110% TCP criteria was SO_4 for the drainable liquids. However, other than those spikes too low to recover, all matrix spikes recovered within the 75%-125% acceptance criteria of the governing QA plan. In general, recoveries on blank spikes were good, except for Cl for the sludges (i.e., 120%) and NO_2 for both the sludges and drainable liquids (i.e., 86% and 88%, respectively). Degradation of the column, most likely caused by insoluble sample matrix components depositing on the column, was observed for all runs which involved Tank BY-108 leach samples.

The leaching/dilution processing blanks provide information on the contamination potentially introduced during the dilution process. Very slight chloride, nitrate, and sulfate blank concentrations were observed. However, these blanks typically have no impact on the reported results, since the anion concentration in the samples is high compared to the blank concentration.

The IC water leach blanks reported in Tables 2.4a through 2.4e have been increased from those reported in the IC data package (see Appendix C). The IC blank is typically calculated using an nominal 1 mL (or 1 g) "equivalent" sample size. However, the drainable liquids were processed using an average 0.2 mL samples size and the sludges were processed using a average 0.5 mL sample size. Therefore, the blank concentrations (or detection limits) reported in the data package have been increased 5-fold for the drainable liquids and 2-fold for the sludges. This provides a better representation of the blank's contribution to the sample concentration. Also, the IC results reported in the data package for the drainable liquid samples are calculated in $\mu g/g$; the results reported in Tables 2.4a through 2.4e have been adjusted for density and reported as $\mu g/mL$.

Total Cyanide Analysis

Sample Preparation and Analysis:

The total cyanide analyses were performed "directly" on homogenized material from Core 99 Segment 1 and Quarter Segments 2A, 2D, 3A, and 4A through 4D, and on diluted drainable liquids from Segment 2 and Segment 3. The sludge and drainable liquid samples were pre-treated and distilled following procedure PNL-ALO-285, "Total Cyanide by Remote Microdistillation and Argentometric Titration". The microdistillations were performed in the SAL and distillates transferred to the Inorganic Analysis Group for subsequent cyanide determination. Total cyanide was determined either by argentometric titration or calorimetrically using a Lachat Autoanalyzer following procedure PNL-ALO-289, "Total Cyanide Determination by Spectrophotometry (Manual or Automated) or Argentometric Titration".

Analytical Results:

The total cyanide results are presented in Tables 2.4a through 2.4e. Since the total CN was anticipated to be very high in Tank BY-108, titrations were initiated on the distillates prepared from the microdistillation operation. The initial samples were titrated, i.e., sludge Segments 1 sample and duplicate and Segment 2A sample and duplicate, and measured essentially no appreciable cyanide. Based on this information, the residual solutions from these samples and the remaining segment samples were analyzed calorimetrically. The results reported from agrentometric titration for sludge Segment 1 sample is an upper estimate only, and since the entire sample was titrated, there is no calorimetric result available. All sludge samples are reported in $\mu g/g$ and the drainable liquids are reported in $\mu g/mL$.

Quality Control, Precision, and Accuracy:

Sample and duplicate results are reported for both drainable liquids and each segment and quarter segment. Distillation blanks, matrix spikes, and spike blanks are also reported for the distillation batch. In addition to the

SAL distillation batch QC samples, an additional distillation was performed by the Inorganic Analysis Group to provide an alternate verification standard to validate the calibration curve established by the calibration standards. For total cyanide, the distilled blank spike is considered the laboratory control standard; this standard recovered at 89% and 88%. Although this recovery is within the 85%-115% acceptance criteria for the governing QA Plan, it exceeds the 90%-110% criteria of the TCP. Besides the control standard, the continuing calibration verification standard failed to meet the 90%-110% TCP accuracy requirement, but is also within the 85%-115% acceptance criteria of the governing QA plan.

The TCP has established a 10% criteria for precision; the RPD between sample and duplicate is used to evaluate the precision of the cyanide analysis. Sludge Segment 1 RPD is unavailable due to the inability of the titration method to accurately measure low cyanide concentration. Also, since Segment 4C sample and Segment 4D duplicate were lost during the microdistillation, no RPDs could be calculated for these segments.

The accuracy of the total cyanide method (i.e., distillation and analysis) is estimated by the use of matrix spikes. Low recoveries on matrix spikes suggest that the reported results may be bias due to matrix interferences. The spike levels were selected based on the expected cyanide content of the tank material; however, in general the tank drainable liquids and sludge contained significantly less cyanide than anticipated. The spike recoveries were reasonably good at 88% for the drainable liquid and 79% for the sludge. Although the spike recovery for the sludge did not meet the TCP 90%-110% criteria, the spike is not considered representative of the samples since the spike is two orders of magnitude above the maximum sample concentration and required very high dilutions to be measured calorimetrically.

The distillation blank provides information regarding cyanide contamination introduced during the distillation and/or measurement process. Except for the Segment 4C sample and the Segment 4D duplicate, which have suspect results, the blank contributed less than 1% to any sample.

Besides the estimate reported for the Segment 1 sample, the results for the Segment 1 duplicate and the Segment 2A sample and duplicate are also reported as estimates, since the initial volume of the distillate and the final volume remaining (after attempts at measuring the cyanide by titration) are not accurately known. The volumes remaining after the attempt at titrating these samples is required to calculate the concentration of cyanide in the aliquots taken for the calorimetric measurement. These volumes have been <u>estimated</u> by weighing; however, errors associated with estimating the volume are not expected to affect the final reported concentration for these segments by more than five to ten percent. Due to the small added uncertainty for Segment 1 and Segment 2A results, and the loss of the Segment 4C sample and 4D duplicate, redistillation and analysis was considered for these segments. However, with the highest cyanide concentration being about 25 times lower than the threshold action limit, no reruns were performed.

Total Organic Carbon, Total Inorganic Carbon, & Total Carbon Analysis

Sample Preparation and Analysis:

The total organic carbon, total inorganic carbon, and total carbon (TOC/TIC/TC) analyses were performed "directly" on homogenized material from Core 99 Segment 1 and Quarter Segments 2A, 2D, 3A, and 4A through 4D, and on diluted drainable liquids from Segment 2 and Segment 3. The TOC/TIC/TC analyses of the core sludge segments and quarter segments were performed by the SAL using a UIC Coulometrics system following procedure PNL-ALO-381, "Determination of TC, TOC, and TIC in Radioactive Liquids, Soils, and Sludges by the Hot Persulfate Method." Dilutions of the drainable liquids were also performed in the SAL and sample aliquots then transferred to the Inorganic Analysis Group for subsequent UV-catalyzed TOC/TIC/TC analysis using a Dohrmann DC80 system following procedure PNL-ALO-382, "Solution Analysis: Carbon."

Analytical Results:

The TOC/TIC/TC analytical results are presented in Tables 2.4a through 2.4e. The drainable liquids are reported in μg/mL and the sludge segments and quarter segments are reported in μg/g. For all TOC/TIC/TC analyses, the results exceed the estimated method detection limit of the method used; that is, 30 μg/g TIC and 50 μg/g TOC for the hot persulfate method, and 50 μg/mL TC or TIC for the UV-catalyzed solution method. For the hot persulfate method, the TIC and TOC are analyzed on each sample and the TC is obtained by summation. For the UV-catalyzed solution method, the TC and TIC are analyzed on each sample and the TOC is obtained by difference.

Quality Control, Precision, and Accuracy:

Sample and duplicate results are reported for both drainable liquids and each segment and quarter segment sample. Processing blanks are also reported for the drainable liquids, since the drainable liquids were diluted by SAL prior to TOC/TIC/TC analysis. However, no processing blanks are reported for the sludge segment samples analyzed by the direct hot persulfate method. This method requires results to be corrected for the average blank carbon levels, as well as the average check standard recoveries. Therefore, all sludge sample results have been corrected for the blank contribution and the check standard recovery. Matrix spikes are included for both the drainable liquid UV-catalyzed analyses and the sludge segment hot persulfate analyses.

Neither the hot persulfate method nor the UV-catalyzed method undergo user calibration for the carbon measurements; the calibration of the instrumentation is established by the manufacturer. Both methods verify instrument response by the use of check standards. The hot persulfate method uses the check standards to adjust the reported result for the average standard recovery. The recoveries demonstrated by the hot persulfate method ranged from 90.5% to 97.1% for TIC and 87% to 96.6% for TOC. Although two TOC standards recovered at less than the 90% recovery required by the TCP, the average TOC recovery used to correct the analytical results was above 90% for each analytical run. Since the analytical results are corrected for the average standard recovery, the demonstrated recovery should not affect the quality of the reported results.

The RPD between duplicates is used to evaluate the precision of the analytical laboratory sample processing and analysis. The majority of the RPD values for sludges (analyzed by the hot persulfate method) meet the TCP 10% criteria. However, a few sludge samples and both drainable liquid samples (analyzed by the UV-catalyzed method) do demonstrate very poor RPDs (i.e., up to 58% for the sludges and 75% for drainable liquids). The poor RPDs for the sludges are attributed primarily to the inconsistency of the sample material and/or the inability to obtain fully representative analytical samples. In the case of TOC for the UV-catalyzed method, the poor RPDs are attributed to the small difference between the TC and TIC sample concentrations.

The accuracy of the TOC/TIC/TC results is estimated by the matrix spike recovery. Low recoveries on matrix spikes provide indications of matrix interferences which may adversely affect the reported analytical results. The hot persulfate TOC/TIC/TC results show excellent recovery for Segment 1, but very low recovery for Quarter Segment 4A. Although the TOC/TIC are very high in Quarter Segment 4A, the TOC/TIC spikes added were 25% above the TOC/TIC measured in the sample, which should have provided adequate spike for determining recovery. Considering this and the fact that the RPD for Quarter Segment 4A is very good, suggests that the low spike recovery is matrix-related (perhaps a high salt content), and that the carbon values reported may be bias low. The UV-catalyzed TOC/TIC/TC sample spike recoveries were quite poor for both TC and TIC, recovering at 130% and 69%, respectively. Since the blank spike recoveries are within the 90%-110% acceptance criteria, the poor spike recoveries on the drainable liquids are attributed to poor analytical reproducibility (i.e., 28% RPD on TIC) and matrix interferences (e.g., possible residual suspended solids after filtering).

The processing blank for the drainable liquid samples analyzed by the UV-catalyzed method provides information on the contamination potential introduced during the dilution process. A very slight TC blank concentration was observed. However, it has no impact on the reported results, since the TC concentration in the samples is very high compared to the blank concentration.

Table 2-4a: BY-108, Core 99 Drainable Liquid, Segment 2 & Segment 3, Anions/Carbon/CN

Segment 2 Drainable Liquid

Analyte	ALO Log #	Sample (µg/mL)	Duplicate (µg/mL)	RPD (%)	Blank (µg/mL)	Smp Spk (%Rec)	Blk Spk (%Rec)
CN	95-07932-G	715	711	<1	0.25	88	88
TOC	95-07932-J	1400	3100	72	<50	--	--
TIC	95-07932-J	8500	6400	28	<50	69	98
TC	95-07932-J	9900	9500	5	64	130	110
Fluoride	95-07932-C	<400	<400	n/a	<35	75	100
Chloride	95-07932-C	3300	3300	0	<35	n/a	100
Nitrite	95-07932-C	57100	56300	1	<70	n/a	88
Nitrate	95-07932-C	230000	236000	2	110	n/a	103
Phosphate	95-07932-C	700	700	0	<70	76	94
Sulfate	95-07932-C	1100	1000	10	80	93	95

Segment 3 Drainable Liquid

Analyte	ALO Log #	Sample (µg/mL)	Duplicate (µg/mL)	RPD (%)	Blank (µg/mL)	Smp Spk (%Rec)	Blk Spk (%Rec)
CN	95-07935-G	521	532	2	--	--	--
TOC	95-07935-J	2400	1100	72	--	--	--
TIC	95-07935-J	7200	8100	11	--	--	--
TC	95-07935-J	9600	9200	4	--	--	--
Fluoride	95-07935-C	<600	<600		--	--	--
Chloride	95-07935-C	3400	3400	0	--	--	--
Nitrite	95-07935-C	60500	61100	1	--	--	--
Nitrate	95-07935-C	204000	226000	10	--	--	--
Phosphate	95-07935-C	1300	1300	0	--	--	--
Sulfate	95-07935-C	1600	1600	0	--	--	--

Notes:
- (a) "<" indicates not detected above reported value.
- (b) RPD = "n/a" when either sample or duplicate are <MDL.
- (c) Smp Spk = "n/a" when Sample result >4X spiking level.
- (d) "--" indicates analysis not performed on sample; e.g., only one Blank, Sample Spike, and Blank Spike analyzed per batch or Blank and Blank Spike not required on TOC/TIC/TC.

Table 2-4b: BY-108, Core 99 Sludge, Segment 1 and Segment 2A, Anions/Carbon/CN

Segment 1 Sludge

Analyte	ALO Log #	Sample (µg/g)	Duplicate (µg/g)	RPD (%)	Blank (µg/g)	Smp Spk (%Rec)	Blk Spk (%Rec)
CN	95-07941-G	<260 (e)	95 (f)	n/a	0.6	79	89
TOC	95-07941-J	1290	920	33	--	94	--
TIC	95-07941-J	1150	1170	2	--	100	--
TC	95-07941-J	2440	2090	15	--	--	--
Fluoride	95-07941-C	6600	5400	20	<20	n/a	100
Chloride	95-07941-C	700	700	0	80	80	120
Nitrite	95-07941-C	8000	8200	2	<60	85	86
Nitrate	95-07941-C	55000	67000	20	160	111	101
Phosphate	95-07941-C	63000	52000	19	<60	n/a	91
Sulfate	95-07941-C	1300	1100	17	120	87	97

Segment 2A Sludge

Analyte	ALO Log #	Sample (µg/g)	Duplicate (µg/g)	RPD (%)	Blank (µg/g)	Smp Spk (%Rec)	Blk Spk (%Rec)
CN	95-07942-G	116 (f)	120 (f)	3	--	--	--
TOC	95-07942-J	890	870	2	--	--	--
TIC	95-07942-J	1470	1590	8	--	--	--
TC	95-07942-J	2360	2460	4	--	--	--
Fluoride	95-07942-C	<500	<500	n/a	--	--	--
Chloride	95-07942-C	800	800	0	--	--	--
Nitrite	95-07942-C	12400	11300	9	--	--	--
Nitrate	95-07942-C	590000	610000	3	--	--	--
Phosphate	95-07942-C	2200	2100	5	--	--	--
Sulfate	95-07942-C	900	900	0	--	--	--

Notes:

(a) "<" indicates not detected above reported value.
(b) RPD = "n/a" when either sample or duplicate are <MDL.
(c) Smp Spk = "n/a" when Sample result >4X spiking level.
(d) "--" indicates analysis not performed on sample; e.g., only one Blank, Sample Spike, and Blank Spike analyzed per batch or Blank and Blank Spike not required on TOC/TIC/TC.
(e) Sample result estimated by titration; see narrative.
(f) Estimated value based on an estimate of final distillate volume used for calorimetric analysis; see narrative.

Table 2-4c: BY-108, Core 99 Sludge, Segment 2D and Segment 3A, Anions/Carbon/CN

Segment 2D Sludge

Analyte	ALO Log #	Sample (µg/g)	Duplicate (µg/g)	RPD (%)	Blank (µg/g)	Smp Spk (%Rec)	Blk Spk (%Rec)
CN	95-07943-G	149	135	10	--	--	--
TOC	95-07943-J	2490	2090	17	--	--	--
TIC	95-07943-J	2340	2480	6	--	--	--
TC	95-07943-J	4830	4570	6	--	--	--
Fluoride	95-07943-C	3900	3800	3	--	--	--
Chloride	95-07943-C	1200	1100	9	--	--	--
Nitrite	95-07943-C	20800	19300	7	--	--	--
Nitrate	95-07943-C	247000	254000	3	--	--	--
Phosphate	95-07943-C	14100	13500	4	--	--	--
Sulfate	95-07943-C	9800	9500	3	--	--	--

Segment 3A Sludge

Analyte	ALO Log #	Sample (µg/g)	Duplicate (µg/g)	RPD (%)	Blank (µg/g)	Smp Spk (%Rec)	Blk Spk (%Rec)
CN	95-07944-G	195	191	2	--	--	--
TOC	95-07944-J	3540	2670	28	--	--	--
TIC	95-07944-J	13100	9100	36	--	--	--
TC	95-07944-J	16700	11700	35	--	--	--
Fluoride	95-07944-C	5600	4800	15	--	--	--
Chloride	95-07944-C	1200	1100	9	--	--	--
Nitrite	95-07944-C	20300	17700	14	--	--	--
Nitrate	95-07944-C	235000	329000	33	--	--	--
Phosphate	95-07944-C	7600	9700	24	--	--	--
Sulfate	95-07944-C	19600	15400	24	--	--	--

Notes:
(a) "<" indicates not detected above reported value.
(b) RPD = "n/a" when either sample or duplicate are <MDL.
(c) Smp Spk = "n/a" when Sample result >4X spiking level.
(d) "--" indicates analysis not performed on sample; e.g., only one Blank, Sample Spike, and Blank Spike analyzed per batch or Blank and Blank Spike not required on TOC/TIC/TC.

Table 2-4d: BY-108, Core 99 Sludge, Segment 4A and Segment 4B, Anions/Carbon/CN

Segment 4A Sludge

Analyte	ALO Log #	Sample (µg/g)	Duplicate (µg/g)	RPD (%)	Blank (µg/g)	Smp Spk (%Rec)	Blk Spk (%Rec)
CN	95-07945-G	742	760	2	--	--	--
TOC	95-07945-J	16500	17100	3	--	80	--
TIC	95-07945-J	13600	12700	6	--	63	--
TC	95-07945-J	30100	29800	1	--	--	--
Fluoride	95-07945-C	8300	5200	46			
Chloride	95-07945-C	1400	1200	15			
Nitrite	95-07945-C	35000	31000	12			
Nitrate	95-07945-C	101000	197000	64			
Phosphate	95-07945-C	19700	13400	38			
Sulfate	95-07945-C	57800	21400	92			

Segment 4B Sludge

Analyte	ALO Log #	Sample (µg/g)	Duplicate (µg/g)	RPD (%)	Blank (µg/g)	Smp Spk (%Rec)	Blk Spk (%Rec)
CN	95-07946-G	1660	1650	1	--	--	--
TOC	95-07946-J	13900	13300	5	--	--	--
TIC	95-07946-J	5090	5530	8	--	--	--
TC	95-07946-J	19000	18800	1	--	--	--
Fluoride	95-07946-C	<600	<500	n/a	--	--	--
Chloride	95-07946-C	1600	1400	13	--	--	--
Nitrite	95-07946-C	48000	44000	9	--	--	--
Nitrate	95-07946-C	69000	65000	6	--	--	--
Phosphate	95-07946-C	32000	46000	36	--	--	--
Sulfate	95-07946-C	6000	4000	40	--	--	--

Notes:

(a) "<" indicates not detected above reported value.

(b) RPD = "n/a" when either sample or duplicate are <MDL.

(c) Smp Spk = "n/a" when Sample result >4X spiking level.

(d) "--" indicates analysis not performed on sample; e.g., only one Blank, Sample Spike, and Blank Spike analyzed per batch or Blank and Blank Spike not required on TOC/TIC/TC.

Table 2-4e: BY-108, Core 99 Sludge, Segment 4C and Segment 4D, Anions/Carbon/CN

Segment 4C Sludge

Analyte	ALO Log #	Sample (µg/g)	Duplicate (µg/g)	RPD (%)	Blank (µg/g)	Smp Spk (%Rec)	Blk Spk (%Rec)
CN	95-07947-G	(e)	1660	n/a	--	--	--
TOC	95-07947-J	5600	4170	29	--	--	--
TIC	95-07947-J	10500	11000	5	--	--	--
TC	95-07947-J	16100	15200	6	--	--	--
Fluoride	95-07947-C	<600	<500	n/a	--	--	--
Chloride	95-07947-C	1400	1300	7	--	--	--
Nitrite	95-07947-C	42000	43000	2	--	--	--
Nitrate	95-07947-C	54000	53000	2	--	--	--
Phosphate	95-07947-C	60000	67000	11	--	--	--
Sulfate	95-07947-C	15000	17000	13	--	--	--

Segment 4D Sludge

Analyte	ALO Log #	Sample (µg/g)	Duplicate (µg/g)	RPD (%)	Blank (µg/g)	Smp Spk (%Rec)	Blk Spk (%Rec)
CN	95-07948-G	1150	(e)	n/a	--	--	--
TOC	95-07948-J	3140	3700	16	--	--	--
TIC	95-07948-J	6050	6910	13	--	--	--
TC	95-07948-J	9190	10600	14	--	--	--
Fluoride	95-07948-C	<500	<400	n/a	--	--	--
Chloride	95-07948-C	1300	1100	17	--	--	--
Nitrite	95-07948-C	40000	34000	16	--	--	--
Nitrate	95-07948-C	50000	42000	17	--	--	--
Phosphate	95-07948-C	79000	78000	1	--	--	--
Sulfate	95-07948-C	21000	9000	80	--	--	--

Notes:

(a) "<" indicates not detected above reported value.
(b) RPD = "n/a" when either sample or duplicate are <MDL.
(c) Smp Spk = "n/a" when Sample result >4X spiking level.
(d) "--" indicates analysis not performed on sample; e.g., only one Blank, Sample Spike, and Blank Spike analyzed per batch or Blank and Blank Spike not required on TOC/TIC/TC.
(e) Distillation prep sheet indicates 95-7947 sample and 95-7948 duplicate lost during distillation.

SECTION 3

RADIOCHEMISTRY

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Radiochemical Analysis

Radiochemical analyses were performed on fusion preparations of samples from Segments 1, 2A, 2D, 3A, 4A, 4B, 4C, and 4D and on acid digestions of the drainable liquids from Segments 2 and 3. In all cases a sample and a duplicate were analyzed; for each batch of samples, a hot cell blank was also analyzed. The fused samples were analyzed for total alpha, total beta, ^{90}Sr, $^{238,239+240}Pu$, uranium, and by gamma energy analysis. The drainable liquids were only analyzed for total alpha, total beta, and by gamma energy analysis. Initially, all of the fusion samples were obtained from the potassium hydroxide fusion preparation. Due to contamination problems noted with one of the blanks for this fusion, analyses were also performed for two samples and a blank prepared by sodium peroxide fusions.

Results for all radiochemical analysis performed on Tank BY-108 are presented in Tables 3-1 through 3-4. It should be noted that both the relative percent difference (RPD) and mean difference (MD) have been calculated. The MD takes individual sample uncertainty into account. Thus, a mean difference less than 1.96 indicates that there is 95% confidence that the sample and duplicate results are in agreement. When the RPD fails but the MD meets this criteria (<1.96) sample precision should be considered acceptable.

Total Alpha Analyses -- Table 3-1: Total alpha analyses were initially performed according to procedures PNL-ALO-420/421. In this method, small aliquots are dried on counting disks and counted using Ludlum scintillation counters. Two problems were noted leading to further analyses. First, the samples for this tank have a very high beta-gamma/alpha ratio. Attempts to make counting plates with reasonable levels of alpha activity resulted in unacceptably high levels of beta-gamma activity. Consequently, samples had to be further diluted resulting in very weak alpha activity and prolonged counting times of 4-8 hours. It was further observed that the counting plates contained residual solids, suggesting probable alpha attenuation due to the residual mass. Although the mass loading due to the sample material was very low (typically < 0.1 mg), solids loading from the fusion flux may have resulted in an alpha absorption effect. Selected samples were reanalyzed using about half the amount of material in an attempt to determine potential mass absorption effects. Since these tests indicated that mass absorption of the alphas was indeed a problem, most of the samples were reanalyzed with and without a matrix spike of a ^{239}Pu standard. The net ^{239}Pu count rate data was then averaged resulting in an average mass absorption correction of 0.722 ± 20%. This factor was applied uniformly to all data for fused samples. No correction was applied to the drainable liquid data since these samples were prepared by acid digestion (no additional salt flux).

The large relative uncertainty for the mass absorption correction is attributable to the loading density on the 1-inch counting plates. This uncertainty more fairly represents the "gross" nature of the alpha measurement from a complex matrix. At the low alpha levels detected in these samples, a 20% uncertainty should be inconsequential. Isotope-specific analyses are needed to obtain more accurate alpha data.

As a check on the quality of the data, an attempt was made to compare the total alpha results with the sum of the Pu and Am measurements. Unfortunately, ^{241}Am data are not available for most of the samples since Am analyses were not requested. Although ^{241}Am can also be detected by gamma energy analysis, positive results were only obtained for three samples. The compton scattering background from the higher-energy, high-activity ^{137}Cs precludes direct gamma measurement of ^{241}Am in the rest of the samples. Hence, there are only three cases where a direct comparison of total alpha and Pu/Am data can be made. In these cases, results are in reasonable agreement; in the other cases it appears that the results are consistent, but not conclusive.

The general agreement between samples and duplicates is poor; however, the uncertainties are quite high for the total alpha data. As discussed below, sample contamination is clearly evident in some of the samples accounting for the high RPD values seen with samples for Segments 1 and 2.

It is also evident that hot cell blanks for the first hot cell preparation batch (95-07941-HCB and 95-07942-H3) indicate significant contamination above the required level of 5% of the sample activity; however, this was not a problem for subsequent hot cell preparation blanks. Sample 95-07942-H3 is the worst case with alpha contamination higher than the sample, but less than the sample duplicate. Consequently, analyses were rerun on the sodium peroxide hot cell fusion preparation, where samples were available. (Such samples have an "N" label, rather than a "H.") The hot cell blank for this preparation (95-07941-N3) indicated no detectable alpha activity. Unfortunately, such samples were not available for sample 95-07942; however, total alpha results for samples 95-07943 and 95-07944 are slightly less than the original fusion preparation results, suggesting that some alpha contamination may be present for these samples.

Plutonium Analyses -- Table 3-1: Plutonium separations and alpha energy analyses were performed for all of the fusion samples following procedures PNL-ALO-423/422. Samples were run in three batches, as indicated in the tables, and standard and matrix spike recoveries for ^{239}Pu were excellent for all three batches. Agreement between samples and duplicates is poor for the first hot cell preparation batch, although agreement is better for the subsequent hot cell preparation batches.

The first hot cell preparation blanks (especially 95-07942-H3) indicate significant plutonium contamination, falling between the sample and duplicate for ^{238}Pu and exceeding both the sample and duplicate for $^{239+240}Pu$. It is further interesting to note that the hot cell contamination appears to be predominately ^{238}Pu whereas the samples themselves are predominately $^{239+240}Pu$. This ratio can thus also be used to gauge the extent of the hot cell contamination. The sodium peroxide hot cell prep blanks indicate a much lower level of contamination and, as with the total alpha, the Pu levels are also lower than the first contaminated hot cell prep samples. Although the hot cell blanks still exceed the 5% criteria even for the sodium peroxide fusion, the absolute alpha activity levels are quite low (< 1 nCi/g). Hence, it might be concluded that it is difficult to measure such low levels of alpha activity for samples prepared in our hot cells due to facility contamination.

Most of the Pu activity appears to be concentrated in Segment 4 where Pu levels are typically 100 times higher than for the other segments. This trend is also mirrored in the total alpha data, although Segment 1 may indicate some contamination problems due to the high 238/239+240 Pu ratio. Hence, although contamination problems are evident for both total alpha and plutonium measurements in Segments 1 and 2, these segments appear to contain only about 1% of the alpha activity contained in the Core 99.

Total Beta Analyses -- Table 3-2: Total beta analyses were performed on all samples using procedures PNL-ALO-430/431. In this method, small aliquots of the prepared samples are dried on counting plates and counted using beta gas flow proportional counters. Due to the much longer range of beta particles, there was no concern regarding mass absorption effects as seen with the total alpha measurements.

Most of the total beta measurements show reasonable agreement between the sample and duplicate except for sample 95-07946. The reasons for this difference are not understood, but may be due to sample heterogeneity, as will be discussed later.

As with the alpha and plutonium data, the first hot cell prep batch also shows significant beta contamination, especially for the blank 95-07942-H3. The sodium peroxide fusion blank shows no beta contamination; however, the total beta results for these samples are in good agreement with the potassium hydroxide fusion samples.

The total beta activity results are in good agreement with the sum of the $^{90}Sr/^{90}Y$ and ^{137}Cs data, as discussed later.

^{90}Sr Analyses -- Table 3-2: ^{90}Sr separations were performed for all of the fusion samples following procedure PNL-ALO-433/431. In this method, ^{90}Sr is separated, dried on counting plates, and counted using beta gas flow proportional counters. A count is taken as soon as possible after the separation and about three days later to measure the ingrowth of the ^{90}Y daughter activity.

The agreement between samples and duplicates is acceptable except for samples 95-07944 and 95-07946. The first hot cell fusion preparation batch again shows significant ^{90}Sr contamination in the blank 95-07942-H3, which is significantly higher than many of the samples. This may account for the high RPD value seen for sample 95-07944. The high RPD for sample 95-07946 may be attributable to sample heterogeneity, as discussed later.

Gamma Energy Analyses -- Table 3-3: Gamma energy analyses were performed for all samples using procedure PNL-ALO-450. Ten milliliters of the sampled prepared in the hot cell were directly counted using germanium gamma detectors. In all cases, the ^{137}Cs activity completely dominated the gamma measurements. ^{241}Am was detected in three samples and $^{154,155}Eu$ was seen in four samples. ^{60}Co was seen in some samples; however, the data are quite erratic suggesting that this might be due to hot cell contamination. ^{40}K was also detected; however, this was not reported since this activity may be due to naturally occurring ^{40}K contained in the potassium hydroxide fusion prep material.

The agreement between samples and duplicates for ^{137}Cs is generally quite good, except for sample 95-07942 (RPD = 32%) where there is known to be significant hot cell contamination, as evidenced in the blank 95-07942-H3 and discussed previously for other analyses. The sodium peroxide hot cell blank indicates no contamination and the samples prepared by this method are in good agreement with the ^{137}Cs results for the potassium hydroxide fusion.

As a check on the quality of the data, the total beta activities were compared to the sum of twice the ^{90}Sr (to account for the ^{90}Y daughter) results plus the ^{137}Cs data. Although each beta emitter has a different efficiency for detection, $^{90}Sr/^{90}Y$ and ^{137}Cs have nearly identical beta counting efficiencies. The good agreement between the total beta activity and the $^{90}Sr/^{90}Y$ and ^{137}Cs activity sum indicates that these three isotopes account for nearly all of the measured beta activity.

Sample 95-07946 appears to show significant heterogeneity effects between the sample and duplicate for both the total beta, ^{90}Sr, and ^{137}Cs results. All three results have high RPD values; however, the total beta activity is in excellent agreement with the sum of the $^{90}Sr/^{90}Y$ and ^{137}Cs activities. This suggests that a real difference between the sample and duplicate exist and may have occurred during the hot cell preparation.

Uranium Analyses -- Table 3-4: Total uranium measurements were performed for all of the fused samples following procedure PNL-ALO-445. In this method, the fluorescence induced by a laser is measured with the sample in solution both before and after the addition of a uranium standard (method of standard addition).

Agreement between samples and duplicates is generally acceptable except for samples 95-07942 where hot cell contamination is quite evident, as discussed previously. Unfortunately, the sodium peroxide hot cell blank also indicates similar levels of uranium contamination. However, it should also be pointed out that most of the uranium is contained in Segment 4 and that the uranium concentrations are at least a factor of 100 times lower in the other segments. As with the alpha problem, this may again be pointing to difficulties in detecting low levels of uranium in samples prepared in our hot cells.

Also, it should be noted that the uranium results from the ICP (see Tables 2-1c and 2-1d) track very favorably with the fluorescence uranium result.

Table 3-1: Tank BY-108, Core 99 Alpha Analysis Results

Sample Number	Prep Batch	*	Total Alpha Analytical Batch	Total Alpha uCi/g	+/- % error	Pu Analytical Batch	238-Pu uCi/g	+/- % error	239+240-Pu uCi/g	+/- % error
Segment 1										
95-07941-H1T	1	F/smp	1	4.22E-02	22	1	2.10E-02	21	5.97E-03	23
95-07941-H2T	1	F/dup	1	1.91E-01	21	1	8.33E-03	8	5.37E-03	8
Mean Difference				1.81			1.42		0.21	
RPD				128 x			86 x		11 x	
95-07941-HCB	1	F/blk	1	7.80E-03	27	1	2.63E-03	11	1.05E-03	14
		std	1	101%		1			98%	
		ms				1			101%	
		blk	1	<9.3E-04		1	<1.7E-04		<1.7E-04	
Segment 2 Drainable Liquid (uCi/mL) **										
95-07932-A1	3	A/smp	2	3.65E-03	29					
95-07932-A2	3	A/dup	2	<1.3E-03						
95-07932-A3	3	A/blk	2	<1.3E-05						
Segment 2A										
95-07942-H1	1	F/smp	1	1.86E-03	47	1	1.22E-03	16	3.53E-04	21
95-07942-H2	1	F/dup	1	7.84E-03	28	1	2.43E-03	11	8.28E-04	15
Mean Difference				1.27			1.83		1.64	
RPD				123 x			66 x		80 x	
95-07942-H3	1	F/blk	1	4.66E-03	27	1	1.52E-03	12	1.45E-03	12
Segment 2D										
95-07943-H1	1	F/smp	1	4.19E-03	25	1	1.15E-03	9	1.43E-03	9
95-07943-H2	1	F/dup	1	2.80E-03	26	1	2.35E-04	13	9.15E-04	11
Mean Difference				0.54			4.24 $		1.58	
RPD				40 x			132 x		44 x	
95-07943-N1	4	FZ/smp	3	2.10E-03	47	3	1.95E-04	16	9.75E-04	12
		std				3			102%	
		blk				3	<1.7E-04		<1.7E-04	
Segment 3 Drainable Liquid (uCi/mL) **										
95-07935-A1	3	A/smp	2	<1.4E-03						
95-07935-A2	3	A/dup	2	<1.4E-03						
Segment 3A										
95-07944-H1	1	F/smp	1	9.29E-03	23	1	1.87E-03	11	3.36E-03	11
95-07944-H2	1	F/dup	1	1.01E-02	23	1	1.60E-03	10	5.02E-03	10
Mean Difference				0.13			0.52		1.33	
RPD				8			16 x		40 x	
95-07944-N1	4	FZ/smp	3	7.99E-03	31	3	4.72E-04	13	3.08E-03	10
05-07941-N3	4	FZ/blk	3	<2.4E-03		3	2.84E-04	34	1.52E-04	37
Segment 4A										
95-07945-H1	2	F/smp	1	1.50E-01	22	2	1.11E-02	14	2.19E-01	9
95-07945-H2	2	F/dup	1	1.74E-01	22	2	3.02E-03	16	1.09E-01	10
Mean Difference				0.24			2.48 $		2.44 $	
RPD				15 x			114 x		67 x	
95-07945-H3	2	F/blk	1	<1.0E-03	15	2	5.04E-04	23	3.19E-04	25
		std				2			103%	
		ms				2			92%	
		blk				2	<1.9E-04		<1.9E-04	
Segment 4B										
95-07946-H1T	2	F/smp	1	3.78E-01	21	2	6.60E-03	15	2.47E-01	9
95-07946-H2T	2	F/dup	1	2.82E-01	21	2	6.06E-03	16	1.94E-01	9
Mean Difference				0.48			0.19		0.94	
RPD				29 x			9		24 x	
95-07946-HCB	2	F/blk	1	7.14E-03	25	2	3.34E-03	11	7.03E-04	15
Segment 4C										
95-07947-H1	2	F/smp	1	4.50E-01	21	2	9.58E-03	14	1.42E-01	10
95-07947-H2	2	F/dup	1	2.93E-01	21	2	7.54E-03	13	1.37E-01	9
Mean Difference				0.70			0.61		0.13	
RPD				42 x			24 x		4	
Segment 4D										
95-07948-H1	2	F/smp	1	3.67E-01	23	2	1.23E-02	13	1.69E-01	10
95-07948-H2	2	F/dup	1	3.02E-01	21	2	9.15E-03	14	1.65E-01	11
Mean Difference				0.31			0.77		0.08	
RPD				19 x			29 x		2	

$ = the mean difference result is greater than or equal to 1.96. there is 95% confidence that the two results are not equal.

* smp = sample, dup = duplicate, blk = methods blank, std = standard
F = KOH - KNO3 Fusion in Ni crucible, FZ = Na2O2 - NaOH Fusion in Zr crucible, A = Acid Digestion
x = RPD value is greater than 10%.
** Only Drainable Liquid samples are reported in uCi/mL.

Table 3-2: Tank BY-108, Core 99 Beta Analysis Results

Sample Number	Prep Batch	*	Total Beta uCi/g	+/- % error	Sr-90 AB	Sr-90 uCi/g	+/- % error
Segment 1							
95-07941-H1T	1	F/smp	2.61E+01	3	1	3.43E+00	8
95-07941-H2T	1	F/dup	2.21E+01	3	1	2.94E+00	8
Mean Difference			1.95			0.68	
RPD			17 x			15 x	
95-07941-HCB	1	F/blk	6.02E-01	10	1	1.19E-01	30
		std			1	104%	
		ms			1	114%	
		blk			1	<3.3E-02	
Segment 2 Drainable Liquid (uCi/mL) **							
95-07932-A1	3	A/smp	1.42E+02	4			
95-07932-A2	3	A/dup	1.44E+02	4			
Mean Difference			0.12				
RPD			1				
95-07932-A3	3	A/blk	1.93E-03	6			
Segment 2A							
95-07942-H1	1	F/smp	2.39E+01	3	1	2.60E-01	16
95-07942-H2	1	F/dup	3.03E+01	3	1	2.70E-01	15
Mean Difference			2.76 $			0.09	
RPD			24 x			4	
95-07942-H3	1	F/blk	1.07E+01	4	1	1.68E+00	9
Segment 2D							
95-07943-H1	1	F/smp	4.94E+01	3	1	2.94E+00	8
95-07943-H2	1	F/dup	4.57E+01	3	1	2.87E+00	8
Mean Difference			0.92			0.11	
RPD			8			2	
95-07943-N1	4	FZ/smp	5.45E+01	3	3	2.83	8
Segment 3 Drainable Liquid (uCi/mL) **							
95-07935-A1	3	A/smp	1.46E+02	4			
95-07935-A2	3	A/dup	1.41E+02	4			
Mean Difference			0.31				
RPD			3				
Segment 3A							
95-07944-H1	1	F/smp	5.23E+01	3	1	5.32E+00	8
95-07944-H2	1	F/dup	6.31E+01	3	1	1.01E+01	8
Mean Difference			2.20 $			2.62 $	
RPD			19 x			62 x	
95-07944-N1	4	FZ/smp	5.15E+01	3	3	6.65E+00	9
05-07941-N3	4	FZ/blk	<9. E-01		3	<3.3E-02	
		std			3	90%	
		blk				<2.4E-02	
Segment 4A							
95-07945-H1	2	F/smp	7.37E+02	4	2	3.38E+02	8
95-07945-H2	2	F/dup	7.21E+02	4	2	3.69E+02	8
Mean Difference			0.19			0.39	
RPD			2			9	
95-07945-H3	2	F/blk	1.30E-01	15	2	<3.3E-02	
		std			2	116%	
		ms			2	96%	
		blk			2	<3.3E-02	
Segment 4B							
95-07946-H1T	2	F/smp	1.22E+03	4	2	5.33E+02	8
95-07946-H2T	2	F/dup	8.41E+02	4	2	3.76E+02	8
Mean Difference			3.20 $			1.50	
RPD			37 x			35 x	
95-07946-HCB	2	F/blk	2.36E-01	11	2	<3.7E-02	
Segment 4C							
95-07947-H1	2	F/smp	2.52E+03	4	2	5.89E+02	8
95-07947-H2	2	F/dup	2.71E+03	4	2	6.62E+02	8
Mean Difference			0.64			0.51	
RPD			7			12 x	
Segment 4D							
95-07948-H1	2	F/smp	3.69E+03	4	2	7.65E+02	8
95-07948-H2	2	F/dup	3.84E+03	4	2	8.56E+02	8
Mean Difference			0.35			0.50	
RPD			4			11 x	

$ = the mean difference result is greater than or equal to 1.96, there is 95% confidence that the two results are not equal.

* smp = sample, dup = duplicate, blk = methods blank, ms = matrix spike, std = standard
F = KOH - KNO3 Fusion in Ni crucible, FZ = Na2O2 - NaOH Fusion in Zr crucible, A = Acid Digestion
x = RPD value is greater than 10%.
** Only Drainable Liquid samples are reported in uCi/mL.

Table 3-3: Tank BY-108, Core 99 Gamma Energy Analysis (GEA) Results

Sample Number	*	Co-60 uCi/g	+/- % Error	Cs-134 uCi/g	+/- % Error	Cs-137 uCi/g	+/- % Error	Eu-154 uCi/g	+/- % Error	Eu-155 uCi/g	+/- % Error	Am-241 uCi/g	+/- % Error
Segment 1													
95-7941-H1T	F/smp	8.49E-03	12	<8.E-03		2.02E+01	3					<3. E-02	
95-7941-H2T	F/dup	<6. E-03		<2.E-02		1.77E+01	3					1.16E-01	15
Mean Difference						1.55							
RPD						13	x						
95-7941-HCB	F/blk	1.77E-03	15	1.67E-03	18	4.30E-01	4					<4. E-03	
95-7941-N3		<6.E-03		<5E-03		3.81E-02	12	<2.E-02		<2.E-02		<2. E-02	
Segment 2 Drainable Liquid (uCi/mL) **													
95-7932-A1	A/smp	<3. E-03		<3. E-02		1.29E+02	6	<6. E-03		<1. E-01		<7. E-02	
95-7932-A2	A/dup	<3. E-03		<3. E-02		1.30E+02	6	<6. E-03		<1. E-01		<7. E-02	
Mean Difference						0.05							
RPD						1							
95-7932-A3	A/blk	<4. E-04		<3. E-04		7.91E-04	35	<6. E-04		<1. E-03		<6. E-04	
Segment 2A													
95-7942-H1	F/smp	<2. E-02		<5.E-02		2.39E+01	5					<1. E-01	
95-7942-H2	F/dup	<3. E-03		<2.E-02		3.30E+01	3					<6. E-02	
Mean Difference						2.93	$						
RPD						32	x						
95-7942-H3	F/blk	1.64E-03	15	<4.E-03		9.36E+00	4					<2. E-02	
Segment 2D													
95-7943-H1	F/smp	1.94E-03	14	<8.E-03		4.82E+01	3					<4. E-02	
95-7943-H2	F/dup	2.83E-03	17	<9.E-03		4.53E+01	3					<3. E-02	
Mean Difference		0.81				0.73							
RPD		37	x			6							
95-7943-N1	F/smp	<6. E-03		<3.E-02		5.23E+01	3	<2.E-02		<8.E-02		<8.E-02	
Segment 3 Drainable Liquid (uCi/mL) **													
95-7935-A1	A/smp	<3. E-03		<4. E-02		1.41E+02	6	<7. E-03		<1. E-01		<7. E-02	
95-7935-A2	A/dup	<3. E-03		<3. E-02		1.37E+02	6	<7. E-03		<1. E-01		<7. E-02	
Mean Difference						0.17							
RPD						3							
Segment 3A													
95-7944-H1	F/smp	3.29E-03	10	<9E-03		4.34E+01	3					<4. E-02	
95-7944-H2	F/dup	2.30E-03	13	<8E-03		4.96E+01	4					<3. E-02	
Mean Difference		1.11				1.31							
RPD		35	x			13	x						
95-7944-N1	FZ/smp	7.82E-3	6	<7E-03		4.33E+1	6	<4.E-03		<2.E-02		<3. E-02	
Segment 4A													
95-7945-H1	F/smp	1.32E-02	3	<5.E-03		7.05E+01	3	3.93E-02	3	3.83E-02	8	1.94E-02	45
95-7945-H2	F/dup	5.44E-03	5	<4.E-03		7.21E+01	3	3.54E-02	3	3.67E-02	8	1.64E-02	40
Mean Difference		8.08	$			0.26		1.23		0.19		0.14	
RPD		83	x			2		10	x	4		17	x
95-7945-H3	F/blk	2.22E-03	9	<6.E-04		6.05E-02	4	<2. E-03		<2. E-03		<2. E-03	
Segment 4B													
95-7946-H1T	F/smp	6.21E-03	14	<2.E-02		1.13E+02	3	8.28E-02	4	8.62E-02	12	<9. E-02	
95-7946-H2T	F/dup	7.75E-03	11	<2.E-02		9.48E+01	3	6.20E-02	5	6.75E-02	14	3.20E-02	65
Mean Difference		0.63				2.06	$	2.29	$	0.67			
RPD		22	x			18	x	29	x	24	x		
95-7946-HCB	F/blk	1.75E-03	16	<1.E-03				<2. E-03		<2. E-03		<3. E-03	
Segment 4C													
95-7947-H1	F/smp	<3. E-02		<4.E-01		1.20E+03	5	<2. E-01		<2. E+00		<8. E-01	
95-7947-H2	F/dup	<3. E-02		<4.E-01		1.34E+03	5	<2. E-01		<2. E+00		<7. E-01	
Mean Difference						0.78							
RPD						11							
Segment 4D													
95-7948-H1	F/smp	<4. E-02		<5.E-01		2.08E+03	5	<2. E-01		<2. E+00		<1. E+00	
95-7948-H2	F/dup	<4. E-02		<5.E-01		2.12E+03	5	<2. E-01		<2. E+00		<2. E+00	
Mean Difference						0.13							
RPD						2							

$ = the mean difference result is greater than or equal to 1.96, there is 95% confidence that the two results are not equal.

* smp = sample, dup = duplicate, blk = methods blank
F = KOH - KNO3 Fusion in Ni crucible, FZ = Na2O2 - NaOH Fusion in Zr crucible, A = Acid Digestion

** Only Drainable Liquid samples are reported in uCi/mL.

Table 3-4: Tank BY-108, Core 99 Uranium Analysis Results

Sample Number	Prep Batch	*	Analytical Batch	Uranium ug/g	+/- % error	
Segment 1						
95-07941-H1T	1	F/smp	1	1.01E+02	14	
95-07941-H2T	1	F/dup	1	1.02E+02	14	
Mean Difference				0.02		
RPD				1		
95-07941-HCB	1	F/blk	1	3.33E+01	35	
		std	1	105%		
		blk	1	< 5. E-02		
Segment 2A						
95-07942-H1	1	F/smp	1	2.20E+01	51	
95-07942-H2	1	F/dup	1	3.05E+01	35	
Mean Difference				0.27		
RPD				32		x
95-07942-H3	1	F/blk	1	1.58E+01	67	
Segment 2D						
95-07943-H1	1	F/smp	1	1.26E+02	5	
95-07943-H2	1	F/dup	1	1.24E+02	5	
Mean Difference				0.11		
RPD				2		
95-07943-N1	4	FZ/smp	3	1.35E+02	5	
		std	3	105%		
		blk	3	<2.6E-02		
Segment 3A						
95-07944-H1	1	F/smp	2	5.01E+02	24	
95-07944-H2	1	F/dup	2	5.35E+02	18	
Mean Difference				0.11		
RPD				7		
		std	2	100%		
		blk	2	<5. E-01		
95-07944-N1	4	FZ/smp	4	4.72E+02	17	
05-07941-N3	4	FZ/blk	3	1.34E+01	67	
		std	4	105%		
		blk	4	<3.3E-01		
Segment 4A						
95-07945-H1	2	F/smp	1	3.23E+04	6	
95-07945-H2	2	F/dup	1	3.19E+04	5	
Mean Difference				0.08		
RPD				1		
95-07945-H3	2	F/blk	1	2.19E+01	48	
Segment 4B						
95-07946-H1T	2	F/smp	2	5.49E+04	21	
95-07946-H2T	2	F/dup	2	4.86E+04	24	
Mean Difference				0.19		
RPD				12		x
95-07946-HCB	2	F/blk	1	<1.1E+01		
Segment 4C						
95-07947-H1	2	F/smp	5	4.67E+04	21	
95-07947-H2	2	F/dup	5	4.82E+04	16	
Mean Difference				0.06		
RPD				3		
		std		103%		
		blk		<3. E-01		
Segment 4D						
95-07948-H1	2	F/smp	2	4.58E+04	26	
95-07948-H2	2	F/dup	1	5.04E+04	5	
Mean Difference				0.19		
RPD				10		

NOTE: If the mean difference result is greater than or equal to 1.96, there is 95% confidence that the two results are not equal.

* smp = sample, dup = duplicate, blk = methods blank, std = standard
F = KOH - KNO3 Fusion in Ni crucible, FZ = Na2O2 - NaOH Fusion in Zr crucible, A = Acid Digestion
x = RPD value is greater than 10%.

WHC-SD-WM-DP-145, REV. 1

SINGLE SHELL TANK

WASTE CHARACTERIZATION PROJECT

TANK BY-108 CORE 99
Revision 0

September 1995

Prepared By: KL Silvers
LR Greenwood
RT Steele
JM Tingey
MW Urie

Pacific Northwest Laboratory

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WHC-SD-WM-DP-145, REV. 1

SINGLE SHELL TANK

WASTE CHARACTERIZATION PROJECT

APPENDICES

PRIMARY ANALYTICAL DATA

TANK BY-108 CORE 99
Revision 0

September 1995

Pacific Northwest Laboratory

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WHC-SD-WM-DP-145, REV. 1

SINGLE SHELL TANK

WASTE CHARACTERIZATION PROJECT

APPENDIX A

SUPPORTING DOCUMENTATION

TANK BY-108 CORE 99

September 1995

Pacific Northwest Laboratory

A1 - SIGNATURE LIST

325 BUILDING STAFF
SIGNATURE LIST

ANALYST NAME	INITIALS	WRITTEN NAME	WRITTEN INITIALS
QA/QC			
KJ KUHL-KLINGER	KJK	[illegible]	[illegible]
OP BREDT	OPB	Delia Bredt	OPB
JD MATHESON	JDM	JD Matheson	JDM
RG SWOBODA	RGS	[illegible]	rgs
PROJ MGMT & SUPPORT			
KL SILVERS	KLS	[illegible]	KLS
BM THORNTON	BMT	[illegible]	[illegible]
DL BELLOFATTO	DLB	Diana L Bellofatto	DLB
TL BURRUSS	TLB	Trina L Burruss	TLB
EA NELSON	EAN	Beth A. Nelson	EAN
LE TOOKER	LET	Lora Tooker	LET
SHIELDED ANALYTICAL LABORATORY			
RT STEELE	RTS	Rick T Steele	RTS
KJ SMITH	KJS	Karla J Smith	KJS
FV HOOPES	FVH	[illegible]	[illegible]
CE CHAMBERLIN	CEC	C E Chamberlin	CEC
LP DARNELL	LPD	Lori P. Darnell	L.P.D.
IC HENRY	ICH	[illegible] C. Henry	ICH
JK RAU	JKR	John K. Rau	JKR
INORGANIC			
MW URIE	MWU	M W Urie	MWU
DL BALDWIN	DLB	D L Baldwin	DLB
BJ COOK	BJC	BJ Cook	[illegible]
PK MELETHIL	PKM	PK Melethil	[illegible]
MM O'NEILL	MMO	M.M. O'Neill	M.M.O.
JJ WAGNER	JJW	[illegible] Wagner	[illegible]
D ORTIZ	DO	D. Ortiz	DO
DR SANDERS	DRS	Danny Sanders	DRS

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325 BUILDING STAFF
SIGNATURE LIST

ANALYST NAME	INITIALS	WRITTEN NAME	WRITTEN INITIALS
ORGANIC			
EW HOPPE	EWH	[illegible]	[illegible]
GS KLINGER	GSK	[illegible]	[illegible]
GA ROSS	GAR	[illegible]	[illegible]
MJ STEELE	MJS	MJ Steele	[illegible]
RADIOCHEMISTRY			
LR GREENWOOD	LRG	LR Greenwood	LRG
SK FADEFF	SDF	Sandra K Fadeff	SKF
RT RATNER	RTR	RTR	RTR
TL TRANG -LE	TLT	T Trang - le	TTL
KA POEPPEL	KAP	Ka Poeppel	[illegible]
KK THOMAS	KKT	K.K. Thomas	K.K.T.
PROCESS CHEMISTRY			
JM TINGEY	JMT	JM Tingey	J T
SM HEINISCH	SMH	SM Heinisch	Smh
PR BREDT	PRB	Paul Bredt	PRB
DL ALEXANDER	DLA	DL Alexander	DLA
GM RICHARDSON	GMR	GM Richardson	GMR
D RINEHART	DR	D.E. [illegible]	D.E.R.
SM TINGEY	SMT	[illegible]	[illegible]

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A2 - WHC CHAINS OF CUSTODY

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DON'T SAY IT -- *Write It!* **Date:** September 28, 1995

To: File (BY-108 Core Analysis) **From:** KL Silvers KLS

Subject: BY-108 SAMPLES RECEIVED WITHOUT CHAIN-OF-CUSTODY

Tank BY-108 Core 99, Segments 1-4 were received from the WHC 222-S Laboratory starting on August 15 and completing on August 25, 1995. All shipments were documented via a Radioactive Shipping Record (RSR). No chain-of-custody documentation accompanied the shipments. Sample identification (i.e. sample number) was communicated by a fax. During the removal of samples from the shipping casks it was noted that the sample numbers for Segment 4A, 4B, 4C, and 4D did not match the previous faxed sample identification information. The WHC point of contact (POC) was contacted and the issue communicated. The POC advised that Segment 4 had been further subsampled (30g) into new containers with new identification numbers. The new numbers were later faxed and the correlation to sample identification completed.

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A3 - PNL CHAINS OF CUSTODY

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HLRF TO SAL CHAINS OF CUSTODY

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Page 1 of 1 **Chain of Custody Number** TWC-45

ACL CHAIN OF CUSTODY

SAMPLE DESCRIPTION Tank BY-108, Core 99, Segment 1 and Qtr Segment 4B ORIGINATOR HLRF

APPLICABLE TEST INSTRUCTION TI95-TWC-01

ANALYSIS REQUESTED OR DEPARTMENT Homogenization Test

PREP METHOD N/A

ACL SAMPLE NUMBER	SAMPLE DESCRIPTION	SENDER	DATE	RECEIVER	DATE
~~95-07941-H1T~~ 95-07941-T-1	BY-108, C99, S1-Top	Scott Timpy	8/29/95	CEC	8-29-95
~~95-07941-H2T~~ 95-07941-T-2	BY-108, C99, S1-Top Dup	Scott T	8/29/95	CEC	8-29-95
~~95-07941-H1B~~ 95-07941-B-1	BY-108, C99, S1-Bottom	Scott T	8/29/95	CEC	8-29-95
~~95-07941-H2B~~ 95-07941-B-2	BY-108, C99, S1-Bottom Dup	Scott T	8/29/95	CEC	8-29-95
~~95-07946-H1T~~ 95-07946-T-1	BY-108, C99, Qtr Seg 4B Top	Scott T	8/29/95	CEC	8-29-95
~~95-07946-H2T~~ 95-07946-T-2	BY-108, C99, Qtr Seg 4B Top Dup	Scott T	8/29/95	CEC	8-29-95
~~95-07946-H1B~~ 95-07946-B-1	BY-108, C99, Qtr Seg 4B Bottom	Scott T	8/29/95	CEC	8-29-95
~~95-07946-H2B~~ 95-07946-B-2	BY-108, C99, Qtr Seg 4B Bottom Dup	Scott T	8/29/95	CEC	8-29-95

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Chain of Custody Number TWC-51

ACL CHAIN OF CUSTODY

SAMPLE DESCRIPTION Tank BY-108, Core 99

ORIGINATOR HLRF

APPLICABLE TEST INSTRUCTION TI95-TWC-02

ANALYSIS REQUESTED OR DEPARTMENT Chemical Analyses per Test Instruction

PREP METHOD None

ACL SAMPLE NUMBER	SAMPLE DESCRIPTION	SENDER	DATE	RECEIVER	DATE
95-07945	7716	Scott R	9-6-95	CEC	9-6-95
95-07946	7717	Scott R	9-6-95		
95-07947	7718	Scott R	9-6-95		
95-07948	7719	Scott R	9-6-95		
95-07935	7398	Scott R	9-6-95		
95-07932	7397	Scott R	9-6-95		
95-07944	7379	Scott R	9-6-95		
95-07942	7506	Scott R	9-6-95		
95-07943	7505	Scott R	9-6-95		
95-07941	7504	Scott R	9-6-95		

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Page 1 of 1 Chain of Custody Number TWC-46

ACL CHAIN OF CUSTODY

SAMPLE DESCRIPTION Tank BY-108, Core 99, Segment 1 ORIGINATOR SAL

APPLICABLE TEST INSTRUCTION TI95-TWC-02

ANALYSIS REQUESTED OR DEPARTMENT Homogenization Test - ICP

PREP METHOD Ni/KOH Fusion

ACL SAMPLE NUMBER	SAMPLE DESCRIPTION	SENDER	DATE	RECEIVER	DATE
95-07941-H1T	BY-108, C99, Segment 1-Top	CEC	9-1-95	DLS	9-1-95
95-07941-H2T	BY-108, C99, Segment 1-Top Dup				
95-07941-HCB	Methods Blank				
95-07941-H1B	BY-108, C99, Segment 1-Bottom				
95-07941-H2B	BY-108, C99, Segment 1- Bottom Dup				

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Chain of Custody Number TWC-47

ACL CHAIN OF CUSTODY

SAMPLE DESCRIPTION Tank BY-108, Core 99, Segment 1 ORIGINATOR SAL

APPLICABLE TEST INSTRUCTION TI95-TWC-02

ANALYSIS REQUESTED OR DEPARTMENT Homogenization Test - GEA

PREP METHOD Ni/KOH Fusion

ACL SAMPLE NUMBER	SAMPLE DESCRIPTION	SENDER	DATE	RECEIVER	DATE
95-07941-H1T	BY-108, C99, Segment 1-Top	CEE	9-1-95	K Poppel	9/1/95
95-07941-H2T	BY-108, C99, Segment 1-Top Dup				
95-07941-HCB	Methods Blank				
95-07941-H1B	BY-108, C99, Segment 1-Bottom				
95-07941-H2B	BY-108, C99, Segment 1- Bottom Dup			KP 9/1/95	KP 9/1/95

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Chain of Custody Number TWC-48

ACL CHAIN OF CUSTODY

SAMPLE DESCRIPTION Tank BY-108, Core 99, Quarter Segment 4B ORIGINATOR SAL

APPLICABLE TEST INSTRUCTION TI95-TWC-02

ANALYSIS REQUESTED OR DEPARTMENT Homogenization Test - ICP

PREP METHOD Ni/KOH Fusion

ACL SAMPLE NUMBER	SAMPLE DESCRIPTION	SENDER	DATE	RECEIVER	DATE
95-07946-H1T	BY-108, C99, Qtr Segment 4B Top	CEC	9-1-95	DKS	9-1-95
95-07946-H2T	BY-108, C99, Qtr Segment 4B Top Dup				
95-07946-HCB	Methods Blank				
95-07946-H1B	BY-108, C99, Qtr Segment 4B Bottom				
95-07946-H2B	BY-108, C99, Qtr Segment 4B Bottom Dup				

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Chain of Custody Number TWC-49

ACL CHAIN OF CUSTODY

SAMPLE DESCRIPTION Tank BY-108, Core 99, Quarter Segment 4B

ORIGINATOR SAL

APPLICABLE TEST INSTRUCTION TI95-TWC-02

ANALYSIS REQUESTED OR DEPARTMENT Homogenization Test - GEA

PREP METHOD Ni/KOH Fusion

ACL SAMPLE NUMBER	SAMPLE DESCRIPTION	SENDER	DATE	RECEIVER	DATE
95-07946-H1T	BY-108, C99, Qtr Segment 4B Top	CEC	9-1-95	[illegible]	9-1-95
95-07946-H2T	BY-108, C99, Qtr Segment 4B Top Dup				
95-07946-HCB	Methods Blank				
95-07946-H1B	BY-108, C99, Qtr Segment 4B Bottom				
95-07946-H2B	BY-108, C99, Qtr Segment 4B Bottom Dup				

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Page 1 of 1 Chain of Custody Number TWC-50

ACL CHAIN OF CUSTODY

SAMPLE DESCRIPTION Tank BY-108, Core 99, Segment 1 and Qtr Segment 4B ORIGINATOR SAL

APPLICABLE TEST INSTRUCTION TI95-TWC-02

ANALYSIS REQUESTED OR DEPARTMENT Homogenization Test - ICP

PREP METHOD HNO3-HCl Acid Digestion

ACL SAMPLE NUMBER	SAMPLE DESCRIPTION	SENDER	DATE	RECEIVER	DATE
95-07941-H1T	BY-108, C99, Segment 1 Top	Cre	9-1-95	DRS	9-1-95
95-07941-H2T	BY-108, C99, Segment 1 Top Dup				
95-07941-HCB	Methods Blank				
95-07946-H1T	BY-108, C99, Qtr Segment 4B Top				
95-07946-H2T	BY-108, C99, Qtr Segment 4B Top Dup				

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ACL CHAIN OF CUSTODY

SAMPLE DESCRIPTION Tank BY-108, Core 99 | ORIGINATOR SAL

APPLICABLE TEST INSTRUCTION TI95-TWC-03

ANALYSIS REQUESTED OR DEPARTMENT ICP

PREP METHOD Water Leach

ACL SAMPLE NUMBER	SAMPLE DESCRIPTION	SENDER	DATE	RECEIVER	DATE
95-07941-C1	BY108, C99, S1	[illegible]	9/8/95	[illegible]	9/8/95
95-07941-C2	BY108, C99, S1, Dup	[illegible]			9/8/95
95-07941-C3	Method Blank	[illegible]			
95-07942-C1	BY108, C99, Qtr Seg 2A	[illegible]			
95-07942-C2	BY108,C99,Qtr Seg 2A Dup	[illegible]			
95-07943-C1	BY108, C99 Qtr Seg 2D	[illegible]			
95-07943-C2	BY108, C99 Qtr Seg 2D Dup	[illegible]			
95-07944-C1	BY108, C99, Qtr Seg 3A	[illegible]			
95-07944-C2	BY108,C99,Qtr Seg 3A Dup	[illegible]			
95-07945-C1	BY108, C99, Qtr Seg 4A	[illegible]			
95-07945-C2	BY108,C99, Qtr Seg 4A Dup	[illegible]			
95-07946-C1	BY108, C99, Qtr Seg 4B	[illegible]			
95-07946-C2	BY108,C99, Qtr Seg 4B Dup	[illegible]			
95-07947-C1	BY108, C99, Qtr Seg 4C	[illegible]			
95-07947-C2	BY108,C99, Qtr Seg 4C Dup	[illegible]			
95-07948-C1	BY108, C99, Qtr Seg 4D	[illegible]			
95-07948-C2	BY108,C99, Qtr Seg 4D Dup	[illegible]			

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Chain of Custody Number TWC-53

ACL CHAIN OF CUSTODY

SAMPLE DESCRIPTION Tank BY-108, Core 99

ORIGINATOR SAL

APPLICABLE TEST INSTRUCTION TI95-TWC-03

ANALYSIS REQUESTED OR DEPARTMENT IC

PREP METHOD Water Leach

ACL SAMPLE NUMBER	SAMPLE DESCRIPTION	SENDER	DATE	RECEIVER	DATE
95-07941-C1	BY108, C99, S1	[signature]	9/2/95	M.M.O	9-7-95
95-07941-C2	BY108, C99, S1, Dup	[signature]			
95-07941-C3	Method Blank	[signature]			
95-07941-C4	Matrix Spike	[signature]			
95-07941-C5	Blank Spike	[signature]			
95-07942-C1	BY108, C99, Qtr Seg 2A	[signature]			
95-07942-C2	BY108,C99,Qtr Seg 2A Dup	[signature]			
95-07943-C1	BY108, C99 Qtr Seg 2D	[signature]			
95-07943-C2	BY108, C99 Qtr Seg 2D Dup	[signature]			
95-07944-C1	BY108, C99, Qtr Seg 3A	[signature]			
95-07944-C2	BY108,C99,Qtr Seg 3A Dup	[signature]			
95-07945-C1	BY108, C99, Qtr Seg 4A	[signature]			
95-07945-C2	BY108,C99, Qtr Seg 4A Dup	[signature]			
95-07946-C1	BY108, C99, Qtr Seg 4B	[signature]			
95-07946-C2	BY108,C99, Qtr Seg 4B Dup	[signature]			
95-07947-C1	BY108, C99, Qtr Seg 4C	[signature]			
95-07947-C2	BY108,C99, Qtr Seg 4C Dup	[signature]			
95-07948-C1	BY108, C99, Qtr Seg 4D	[signature]			
95-07948-C2	BY108,C99, Qtr Seg 4D Dup	[signature]			

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Chain of Custody Number TWC-54

ACL CHAIN OF CUSTODY

SAMPLE DESCRIPTION Tank BY-108, Core 99

ORIGINATOR SAL

APPLICABLE TEST INSTRUCTION TI95-TWC-03

ANALYSIS REQUESTED OR DEPARTMENT ICP

PREP METHOD Ni/KOH Fusion

ACL SAMPLE NUMBER	SAMPLE DESCRIPTION	SENDER	DATE	RECEIVER	DATE
95-07941-H1T	BY108, C99, S1 Top	[signature]	9/12/95	DKS	9/12/95
95-07941-H2T	BY108, C99, S1 Top Dup	[signature]			
95-07941-HCB	Method Blank	[signature]			
95-07942-H1	BY108,C99, Qtr Seg 2A	[signature]			
95-07942-H2	BY108,C99,Qtr Seg 2A Dup	[signature]			
95-07942-H3	Method Blank	[signature]			
95-07943-H1	BY108,C99,Qtr Seg 2D	[signature]			
95-07943-H2	BY108,C99,Qtr Seg 2D Dup	[signature]			
95-07944-H1	BY108,C99 Qtr Seg 3A	[signature]			
95-07944-H2	BY108,C99,Qtr Seg 3A Dup	[signature]			
SRM-2709-H	Standard Reference Material	[signature]			

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Page 1 of 1 **Chain of Custody Number TWC-55**

ACL CHAIN OF CUSTODY

SAMPLE DESCRIPTION Tank BY-108, Core 99 ORIGINATOR SAL

APPLICABLE TEST INSTRUCTION TI95-TWC-03

ANALYSIS REQUESTED OR DEPARTMENT ICP

PREP METHOD Ni/KOH Fusion

ACL SAMPLE NUMBER	SAMPLE DESCRIPTION	SENDER	DATE	RECEIVER	DATE
95-07945-H1	BY108, C99, Qtr Seg 4A	[illegible]	9/12/95	[illegible]	9/12/95
95-07945-H2	BY108,C99,Qtr Seg 4A Dup				
95-07945-H3	Method Blank				
95-07946-H1T	BY108,C99,Qtr Seg 4B Top				
95-07946-H2T	BY108,C99,Qtr Seg 4B Top Dup	[illegible]			
95-07946-HCB	Method Blank				
95-07947-H1	BY108,C99,Qtr Seg 4C				
95-07947-H2	BY108,C99,Qtr Seg 4C Dup				
95-07948-H1	BY108,C99 Qtr Seg 4D				
95-07948-H2	BY108,C99,Qtr Seg 4D Dup				
SRM-2709-H	Standard Reference Material	[illegible]			

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ACL CHAIN OF CUSTODY

SAMPLE DESCRIPTION Tank BY-108, Core 99 | ORIGINATOR SAL

APPLICABLE TEST INSTRUCTION TI95-TWC-03

ANALYSIS REQUESTED OR DEPARTMENT ICP

PREP METHOD Zr/Na202 Fusion

ACL SAMPLE NUMBER	SAMPLE DESCRIPTION	SENDER	DATE	RECEIVER	DATE
95-07941-N1	BY108, C99, Segment 1	[illegible]	9/12/95	DKS	9/12/95
95-07941-N2	BY108,C99, Segment 1 Dup				
95-07941-N3	Method Blank				
~~95-07942-N1~~	~~BY108, C99, Qtr Seg 2A~~	RS 9/11-95			
95-07943-N1	BY108, C99, Qtr Seg 2D				
95-07944-N1	BY108, C99, Qtr Seg 3A				
95-07945-N1	BY108, C99, Qtr Seg 4A				
95-07946-N1	BY108, C99, Qtr Seg 4B				
95-07947-N1	BY108, C99, Qtr Seg 4C				
95-07948-N1	BY108, C99, Qtr Seg 4D				
SRM-2709-N	Standard Reference Material				

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Chain of Custody Number TWC-57

ACL CHAIN OF CUSTODY

SAMPLE DESCRIPTION Tank BY-108, Core 99

ORIGINATOR SAL

APPLICABLE TEST INSTRUCTION TI95-TWC-03

ANALYSIS REQUESTED OR DEPARTMENT ICP-Drainable Liquid

PREP METHOD Acid Digest

ACL SAMPLE NUMBER	SAMPLE DESCRIPTION	SENDER	DATE	RECEIVER	DATE
95-07932-A1	BY108, C99, Segment 1	[illegible]	9/11/95	[illegible]	9/11/95
95-07932-A2	BY108,C99, Segment 1 Dup				
95-07932-A3	Method Blank				
95-07932-A4	Matrix Spike				
95-07932-A5	Spike Control				
95-07935-A1	BY108, C99, Segment 3				
95-07935-A2	BY108,C99, Segment 3 Dup				

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Chain of Custody Number TWC-58

ACL CHAIN OF CUSTODY

SAMPLE DESCRIPTION Tank BY-108, Core 99

ORIGINATOR SAL

APPLICABLE TEST INSTRUCTION TI95-TWC-03

ANALYSIS REQUESTED OR DEPARTMENT IC-Drainable Liquid

PREP METHOD Water Leach

ACL SAMPLE NUMBER	SAMPLE DESCRIPTION	SENDER	DATE	RECEIVER	DATE
95-07932-C1	BY108, C99, Segment 1	[illegible]	9/8/95	mmo	9-8-95
95-07932-C2	BY108,C99, Segment 1 Dup	[illegible]			
95-07932-C3	Method Blank	[illegible]			
95-07932-C4	Matrix Spike	[illegible]			
95-07932-C5	Spike Control	[illegible]			
95-07935-C1	BY108, C99, Segment 3	[illegible]			
95-07935-C2	BY108,C99, Segment 3 Dup	[illegible]			

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Chain of Custody Number TWC-59

ACL CHAIN OF CUSTODY

SAMPLE DESCRIPTION Tank BY-108, Core 99

ORIGINATOR SAL

APPLICABLE TEST INSTRUCTION TI95-TWC-03

ANALYSIS REQUESTED OR DEPARTMENT ICP-Drainable Liquid

PREP METHOD Water Leach

ACL SAMPLE NUMBER	SAMPLE DESCRIPTION	SENDER	DATE	RECEIVER	DATE
95-07932-C1	BY108, C99, Segment 1	[illegible]	9/8/95	[illegible]	9/8/95
95-07932-C2	BY108,C99, Segment 1 Dup	[illegible]			
95-07932-C3	Method Blank	[illegible]			
95-07935-C1	BY108, C99, Segment 3	[illegible]			
95-07935-C2	BY108,C99, Segment 3 Dup	[illegible]			

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Chain of Custody Number TWC-60

ACL CHAIN OF CUSTODY

SAMPLE DESCRIPTION Tank BY-108, Core 99

ORIGINATOR SAL

APPLICABLE TEST INSTRUCTION TI95-TWC-03

ANALYSIS REQUESTED OR DEPARTMENT Total Alpha, Total Beta, GEA - Drainable Liquid

PREP METHOD Acid Digest

ACL SAMPLE NUMBER	SAMPLE DESCRIPTION	SENDER	DATE	RECEIVER	DATE
95-07932-A1	BY108, C99, Segment 1	[illegible]	9/11/95	[illegible]	9/11/95
95-07932-A2	BY108,C99, Segment 1 Dup	[illegible]		[illegible]	
95-07932-A3	Method Blank	[illegible]		[illegible]	
95-07935-A1	BY108, C99, Segment 3	[illegible]		[illegible]	
95-07935-A2	BY108,C99, Segment 3 Dup	[illegible]		[illegible]	

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Chain of Custody Number TWC-61

ACL CHAIN OF CUSTODY

SAMPLE DESCRIPTION Tank BY-108, Core 99

ORIGINATOR SAL

APPLICABLE TEST INSTRUCTION TI95-TWC-03

ANALYSIS REQUESTED OR DEPARTMENT Total Alpha, Alpha/AEA, GEA, Total Beta, Sr-90, U/Laser

PREP METHOD Ni/KOH Fusion

ACL SAMPLE NUMBER	SAMPLE DESCRIPTION	SENDER	DATE	RECEIVER	DATE
95-07941-H1T	BY108, C99, Segment 1	[illegible]	9/12/95	Kp	9/12/95
95-07941-H2T	BY108,C99, Segment 1 Dup				
95-07941-HCB	Method Blank				
95-07942-H1	BY108, C99, Qtr Seg 2A				
95-07942-H2	BY108,C99,Qtr Seg 2A Dup				
95-07942-H3	Method Blank				
95-07943-H1	BY108, C99, Qtr Seg 2D				
95-07943-H2	BY108,C99,Qtr Seg 2D Dup				
95-07944-H1	BY108, C99, Qtr Seg 3A				
95-07944-H2	BY108,C99,Qtr Seg 3A Dup				

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Chain of Custody Number TWC-62

ACL CHAIN OF CUSTODY

SAMPLE DESCRIPTION Tank BY-108, Core 99

ORIGINATOR SAL

APPLICABLE TEST INSTRUCTION TI95-TWC-03

ANALYSIS REQUESTED OR DEPARTMENT Total Alpha, Alpha/AEA, GEA, Total Beta, Sr-90, U/Laser

PREP METHOD Ni/KOH Fusion

ACL SAMPLE NUMBER	SAMPLE DESCRIPTION	SENDER	DATE	RECEIVER	DATE
95-07945-H1	BY108, C99, Qtr Seg 4A	[illegible]	9/12/95	[illegible]	9/12/95
95-07945-H2	BY108,C99,Qtr Seg 4A Dup				
95-07945-H3	Method Blank				
95-07946-H1T	BY108, C99, Qtr Seg 4B				
95-07946-H2T	BY108,C99,Qtr Seg 4B Dup				
95-07946-HCB	Method Blank				
95-07947-H1	BY108, C99, Qtr Seg 4C				
95-07947-H2	BY108,C99,Qtr Seg 4C Dup				
95-07948-H1	BY108, C99, Qtr Seg 4D				
95-07948-H2	BY108,C99,Qtr Seg 4D Dup				

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Chain of Custody Number TWC-63

ACL CHAIN OF CUSTODY

SAMPLE DESCRIPTION Tank BY-108, Core 99

ORIGINATOR SAL

APPLICABLE TEST INSTRUCTION TI95-TWC-03

ANALYSIS REQUESTED OR DEPARTMENT Cyanide - Drainable Liquid

PREP METHOD Microdistillation

ACL SAMPLE NUMBER	SAMPLE DESCRIPTION	SENDER	DATE	RECEIVER	DATE
95-07932-G1	BY108, C99, Seg 2 Drainable Liquid	[illegible]	9-14-95	MMO	9-14-95
95-07932-G2	BY108,C99, Seg 2 Drainable Liquid Dup				
95-07932-G3	Method Blank				
95-07932-G4	Matrix Spike				
95-07932-G5	Spike Control				
95-07935-G1	BY108, C99, Seg 3 Drainable Liquid				
95-07935-G2	BY108, C99, Seg 3 Drainable Liquid Dup				

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Chain of Custody Number TWC-64

ACL CHAIN OF CUSTODY

SAMPLE DESCRIPTION Tank BY-108, Core 99

ORIGINATOR SAL

APPLICABLE TEST INSTRUCTION TI95-TWC-03

ANALYSIS REQUESTED OR DEPARTMENT Cyanide - Solids

PREP METHOD Microdistillation

ACL SAMPLE NUMBER	SAMPLE DESCRIPTION	SENDER	DATE	RECEIVER	DATE
95-07941-G1	BY108, C99, Seg 1	[illegible]	9/14/95	MMO	9-14-95
95-07941-G2	BY108,C99, Seg 1 Dup				
95-07941-G3	Method Blank				
95-07941-G4	Matrix Spike				
95-07941-G5	Spike Control				
95-07942-G1	BY108, C99, Seg 2A	[illegible]			
95-07942-G2	BY108, C99, Seg 2A				
95-07943-G1	BY108, C99, Seg 2D				
95-07943-G2	BY108, C99, Seg 2D Dup				
95-07944-G1	BY108, C99, Seg 3A				
95-07944-G2	BY108, C99, Seg 3A Dup	[illegible]			
95-07945-G1	BY108, C99, Seg 4A				
95-07945-G2	BY108, C99, Seg 4A Dup				
95-07946-G1	BY108, C99, Seg 4B				
95-07946-G2	BY108, C99, Seg 4B Dup				
95-07947-G1	BY108, C99, Seg 4C				
95-07947-G2	BY108, C99, Seg 4C Dup				
95-07948-G1	BY108, C99, Seg 4D				
95-07948-G2	BY108, C99, Seg 4D Dup	[illegible]			

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A4 - TEST INSTUCTIONS

TANK CHARACTERIZATION
TEST INSTRUCTIONS FOR HLRF RECEIPT AND ANALYSES OF SST 241-BY-108, CORE 99 SEGMENTS 1-4

DATE PREPARED: August 23, 1995 PREPARED BY: KL Silvers

SAMPLE NUMBERS: 95-07931 - 95-07948

APPROVED BY: [signature] DATE: 8/25/95

CONTROLLING DOCUMENTS: Project No: 21372

WHC TCP: Tank 241-BY-108 Tank Characterization Plan (WHC-SD-WM-TP-275)
Revision OE - ECN No: 6621348 (August, 1995)
PNL QAP: MCS-033, Revision 2 (April, 1995)
Controlling Procedure: PNL-ALO-010

INTRODUCTION

This Test Instruction (TI) defines the scope of work to be completed on Single-Shell Tank (SST) 241-BY-108, Core 99 Segments 1, 2, 3 and 4. The samples from this tank will be analyzed according to TCP (WHC-SD-WM-TP-275 Rev. OE).

The subsampled tank material from WHC will be analyzed as-received for TGA (on unhomogenized tank material subsamples and an aliquot from each of the two drainable liquid samples). In addition, DSC and bulk density will be required on the homogenized samples.

Homogenization will be conducted on all non-TGA sample material. A homogenization check will be performed on Segment 1 and Quarter-Segment 4B. An aliquot of homogenized sample material will be removed and transferred to the Shielded Analytical Laboratory (SAL) for preparation of the homogenization check samples. An ICP and GEA analysis will be conducted to evaluate the homogenization process. If the analytical results indicate the sample to be non-homogenized, another attempt will be made to homogenize prior to performing any analytical determination.

Homogenized sample material will be transferred to the SAL for preparation and distribution to the analytical laboratories to complete the scope of work.

Sample receiving and homogenization activities should be charged against work package K28859 and K28860, respectively.

All activities identified in this Test Instruction shall be charged against Project 21372. All analyses are to be completed at Impact Level 2. All analyses are to be completed following the identified procedures. Any deviations to the procedures must be documented and this documentation must accompany the analytical data. All analytical data are returned to the Laboratory Support Office.

REQUESTED ANALYSES

Requested Analysis		Procedure Number	WP#
TGA	unhomogenized sample	PNL-ALO-508	K28861
DSC	homogenized subsample	PNL-ALO-508	K28861
Bulk Density	homogenized subsample	PNL-ALO-501	K28865

A-HOT CELL SAMPLE INSTRUCTIONS

- All analyses should be performed in duplicate if adequate sample is available. If adequate sample is not available, document and notify the PM.
- Collect a TGA sample aliquot from each of the two drainable liquid samples (i.e. Segment 2 and Segment 3).
- Perform TGA analyses on the unhomogenized solid and liquid samples.
- All other samples should be homogenized. In the event insufficient sample exist to perform the traditional homogenization, a best effort should be made to thoroughly mix the material prior to analysis.
- A homogenization check will be conducted on Segment 1 and Quarter-Segment "B" from Segment 4 (i.e. 4B). Collect duplicate homogenization check samples from the top and bottom (approx. 3 grams) and transfer to the SAL. No further testing shall be conducted until results of the homogenization check are reviewed and results deemed valid. (handwritten: duplicate KLS 8/25/95)
- The ACL sample numbers should be assigned as follows:

CORE IDENTIFICATION	ACL NUMBER
Core 99 - Segment 1 (TGA-unhomogenized)	95-07931
Core 99 - Segment 1	95-07941
Core 99 - Segment 2 (liquid and subsample liquid for TGA)	95-07932
Core 99 - Quarter-Segment 2A (TGA-unhomogenized)	95-07933
Core 99 - Quarter-Segment 2D (TGA-unhomogenized)	95-07934
Core 99 - Quarter-Segment 2A (homogenized)	95-07942
Core 99 - Quarter-Segment 2D "	95-07943
Core 99 - Segment 3 (liquid and subsample liquid for TGA)	95-07935

CORE IDENTIFICATION	ACL NUMBER
Core 99 - Quarter-Segment 3A (TGA-unhomogenized)	95-07936
Core 99 - Quarter-Segment 3A (homogenized)	95-07944
Core 99 - Quarter-Segment 4A (TGA-unhomogenized)	95-07937
Core 99 - Quarter-Segment 4B (TGA-unhomogenized)	95-07938
Core 99 - Quarter-Segment 4C (TGA-unhomogenized)	95-07939
Core 99 - Quarter-Segment 4D (TGA-unhomogenized)	95-07940

CORE IDENTIFICATION	ACL NUMBER
Core 99 - Quarter-Segment 4A (homogenized)	95-07945
Core 99 - Quarter-Segment 4B "	95-07946
Core 99 - Quarter-Segment 4C "	95-07947
Core 99 - Quarter-Segment 4D "	95-07948

Tank BY-108 Core 99 Sample Identification

	WHC Sample Number	ACL Number	Sample Description	Sample Volume
Segment 1	Vial # 7313	95-07931	Unhomogenized TGA Sample	3.3 g
	Jar # 7504	95-07941	Segment 1 Solids	53.1 g
Segment 2	Jar # 7397	95-07932	Drainable Liquid	230 mL
	Jar # 7397	95-07932	Drainable Liquid - TGA Subsample	TBD
	Vial # 7315	95-07933	QS-2A TGA Sample	1.9 g
	Vial # 7314	95-07934	QS-2D TGA Sample	4.0 g
	Jar # 7506	95-07942	QS-2A Solids	7.1 g
	Jar # 7505	95-07943	QS-2D Solids	24.0 g
Segment 3	Jar # 7398	95-07935	Drainable Liquid	25 mL
	Jar # 7398	95-07935	Drainable Liquid - TGA Subsample	TBD
	Vial # 7316	95-07936	QS-3A TGA Sample	3.5 g
	Jar # 7379	95-07944	QS-3A Solids	18.9 g
Segment 4	Vial # 7322	95-07937	QS-4A TGA Sample	4.0 g
	Vial # 7319	95-07938	QS-4B TGA Sample	4.3 g
	Vial # 7318	95-07939	QS-4C TGA Sample	4.6 g
	Vial # 7317	95-07940	QS-4D TGA Sample	5.3 g
	Jar # 7478	95-07945	QS-4A Solids	TBD
	Jar # 7476	95-07946	QS-4B Solids	TBD
	Jar # 7400	95-07947	QS-4C Solids	TBD
	Jar # 7399	95-07948	QS-4D Solids	TBD

TANK CHARACTERIZATION TEST INSTRUCTIONS FOR HOMOGENIZATION CHECK OF SST 241-BY-108, CORE 99 SEGMENT 1 AND QUARTER-SEGMENT 4B

DATE PREPARED: August 23, 1995   PREPARED BY: KL Silvers

SAMPLE NUMBERS: 95-07941 & 95-07946

APPROVED BY: [signature]   DATE: 8/25/95

CONTROLLING DOCUMENTS: Project No: 21372

WHC TCP: Tank 241-BY-108 Tank Characterization Plan (WHC-SD-WM-TP-275)
Revision OE - ECN No: 6621348 (August, 1995)
PNL QAP: MCS-033, Revision 2 (April, 1995)
Controlling Procedure: PNL-ALO-010

INTRODUCTION

This Test Instruction (TI) defines the scope of work to be completed on Single-Shell Tank (SST) 241-BY-108, Core 99 Segment 1 and Quarter-Segment 4B homogenization check samples. The samples from this tank will be analyzed according to TCP (WHC-SD-WM-TP-275 Rev. OE).

All activities identified in this Test Instruction shall be charged against Project 21372. All analyses are to be completed at Impact Level 2. All analyses are to be completed following the identified procedures. Any deviations to the procedures must be documented and this documentation must accompany the analytical data. All analytical data are returned to the Laboratory Support Office.

SAL SAMPLE PREPARATION FOR HOMOGENIZATION CHECK:

Duplicate fusions should be completed for each of the two homogenization check samples. The samples will be distributed for both ICP and GEA. A single Methods Blank is required per batch.

Caustic Fusion   (WPkg K28863)   PNL-ALO-115
(QC Requirements: Duplicate, Blank)

Perform duplicate acid digestions via PNL-ALO-129 for visual confirmation of sample solubility.

REQUESTED ANALYSES FOR HOMOGENIZATION CHECK:

Requested Analysis	Procedure Number	Task Leader	WP#
ICP (Fusion)	PNL-ALO-211	Urie	K28866
GEA (Fusion)	PNL-ALO-450	Greenwood	K28872

SAMPLE IDENTIFICATION SYSTEM FOR HOMOGENIZATION CHECK:

Segment 1	Quarter-Segment 4B	
95-07941-H1T	95-07946-H1T	Sample - Top
95-07941-H2T	95-07946-H2T	Duplicate - Top
95-07941-HCB	95-07946-HCB	Methods Blank
95-07941-H1B	95-07946-H1B	Sample - Bottom
95-07941-H2B	95-07946-H2B	Duplicate - Bottom

TANK CHARACTERIZATION
TEST INSTRUCTIONS FOR ANALYSES OF
SST 241-BY-108, CORE 99 SEGMENTS 1-4

DATE PREPARED: September 12, 1995 PREPARED BY: KL Silvers

SAMPLE NUMBERS: 95-07931 - 95-07948

APPROVED BY: [signature] DATE: 9/12/95

CONTROLLING DOCUMENTS: Project No: 21372

WHC TCP: Tank 241-BY-108 Tank Characterization Plan (WHC-SD-WM-TP-275)
Revision OE - ECN No: 6621348 (August, 1995)
PNL QAP: MCS-033, Revision 2 (April, 1995)
Controlling Procedure: PNL-ALO-010

INTRODUCTION

This Test Instruction (TI) defines the scope of work to be completed on Single-Shell Tank (SST) 241-BY-108, Core 99 Segments 1, 2, 3 and 4. The samples from this tank will be analyzed according to TCP (WHC-SD-WM-TP-275 Rev. OE).

This TI contains instructions for the preparation and analyses by the ACL of all segments, quarter-segments, and drainable liquids from Core 99.

All activities identified in this Test Instruction shall be charged against Project 21372. All analyses are to be completed at Impact Level 2. All analyses are to be completed following the identified procedures. Any deviations to the procedures must be documented and this documentation must accompany the analytical data. All analytical data are returned to the Laboratory Support Office.

SAL SAMPLE PREPARATION INSTRUCTIONS FOR SEGMENTS AND QUARTER-SEGMENTS:

The following tank material samples were transferred from the HLRF:

CORE IDENTIFICATION	ACL NUMBER
Core 99 - Segment 1 (homogenized, mechanical)	95-07941
Core 99 - Segment 2 (drainable liquid)	95-07932
Core 99 - Quarter-Segment 2A (homogenized, manual)	95-07942
Core 99 - Quarter-Segment 2D (homogenized, manual)	95-07943
Core 99 - Segment 3 (drainable liquid)	95-07935
Core 99 - Quarter-Segment 3A (homogenized, manual)	95-07944

CORE IDENTIFICATION	ACL NUMBER
Core 99 - Quarter-Segment 4A (homogenized, mechanical)	95-07945
Core 99 - Quarter-Segment 4B (homogenized, mechanical)	95-07946
Core 99 - Quarter-Segment 4C (homogenized, mechanical)	95-07947
Core 99 - Quarter-Segment 4D (homogenized, mechanical)	95-07948

The following sample preparation steps are required:

Method		Procedure
Acid Digestion for ICP - liquid (QC Requirements: Duplicate, Blank, Spike, Spike Control)	(WPkg K28862)	PNL-ALO-128
Caustic Fusion - Na_2O_2 (QC Requirements - ICP : 1 Duplicate per batch, Blank, Post Digestion Spike, LCS per prep)	(WPkg K28863)	PNL-ALO-114
Caustic Fusion - KOH (QC Requirements - Radchem: Duplicate, Blank) (QC Requirements: Duplicate, Blank, Post Digestion Spike, LCS per prep)	(WPkg K28863)	PNL-ALO-115
Water Leach (QC Requirements - IC: Duplicate, Blank, Spike, Spike Control) (QC Requirements - ICP: Duplicate, Blank)	(WPkg K28864)	PNL-ALO-103
Total CN Prep (QC Requirements: Duplicate, Blank, Spike, Spike Control)	(WPkg K28868)	PNL-ALO-285

REQUESTED ANALYSES:

Requested Analysis	Procedure Number	Task Leader	WP#
ICP (Acid, Fusion, Water Leach)	PNL-ALO-211	Urie	K28866
IC (Water Leach)	PNL-ALO-212	Urie	K28867
CN (Direct)	PNL-ALO-285	Urie	K28868
TOC by Hot Persulfate (Direct - solids)	PNL-ALO-381	Steele	K28869
TOC (Direct - liquids)	PNL-ALO-382	Urie	K28869
Total Alpha (Fusion, Acid)	PNL-ALO-421	Greenwood	K28870
AEA (Pu-239/240) (Fusion)	PNL-ALO-422/423	Greenwood	K28871

Requested Analysis	Procedure Number	Task Leader	WP#
GEA (Fusion, Acid)	PNL-ALO-450	Greenwood	K28872
Total Beta (Fusion, Acid)	PNL-ALO-430/431	Greenwood	K28873
Sr-90 (Fusion)	PNL-ALO-433/431	Greenwood	K28874
U-Laser (Fusion)	PNL-ALO-445	Greenwood	K28875

SAMPLE IDENTIFICATION SYSTEM FOR SEGMENT AND QUARTER-SEGMENTS:

The following sample identification system will be followed. All samples will be idenitified as 95-XXXXX-Y#, where 95-XXXXX is the ACL number. The "Y" letter will identify a Sample Preparation Method. The "#" identifies replicate analyses using the sample preparation method (i.e., duplicates, spikes, blanks, etc.). The Sample Preparation Methods codes are:

A Acid Digestion
C Water Leach
D Direct
G Cyanide Sample Prep
H Caustic Fusion - KOH
J Carbon
N Caustic Fusion - Na_2O_2

The replicate analysis codes are:

1 Sample
2 Sample Duplicate
3 Methods Blank
4 Matrix Spike
5 LCS / Blank Spike
6 Post Digestion Spike

CORE 99 SEGMENT 1 ANALYSES

Analysis	Segment 1 Unhomogenized	Segment 1	
TGA	95-07931		Run in Duplicate
Water Leach		95-07941-C1	Water Leach Sample (IC, ICP)
		95-07941-C2	Water Leach Duplicate
		95-07941-C3	Methods Blank (one per batch)
		95-07941-C4	Spike (IC Only) (one per batch)
		95-07941-C5	Blank Spike (IC Only) (one per batch)
Direct		95-07941-D1	DSC & Density Sample
		95-07941-D2	DSC & Density Duplicate
		95-07941-D3	Methods Blank (one per batch)
Total Cyanide		95-07941-G1	Total CN Sample
		95-07941-G2	Total CN Duplicate
		95-07941-G3	Methods Blank (one per batch)
		95-07941-G4	Matrix Spike (one per batch)
		95-07941-G5	Blank Spike (one per batch)
Homogenization Test Fusion Dissolution KOH		95-07941-H1T	Homogenization Test Fusion Sample - Top *
		95-07941-H2T	Homogenization Test Fusion Duplicate - Top
		95-07941-HCB	Homogenization Test Fusion Blank
		95-07941-H1B	Homogenization Test Fusion Sample - Bottom
		95-07941-H2B	Homogenization Test Fusion Duplicate - Bottom
Fusion Dissolution KOH		95-07941-H1	KOH Fusion Sample (ICP, Radchem**)
		95-07941-H2	KOH Fusion Duplicate
		95-07941-H3	Methods Blank (one per batch)
		95-07941-H5	LCS (one per prep)
		95-07941-H6	Post Digestion Spike (ICP Only)
Carbon		95-07941-J1	Carbon Analysis Sample (TIC/TOC/TC)
		95-07941-J2	Carbon Analysis Duplicate
		95-07941-J3	Methods Blank (one per batch)
		95-07941-J4	Matrix Spike (one per batch)
Fusion Dissolution Na2O2		95-07941-N1	Na2O2 Fusion Sample (ICP)
		95-07941-N2	Na2O2 Fusion Duplicate (one per batch)
		95-07941-N3	Methods Blank (one per batch)
		95-07941-N5	LCS (one per prep)
		95-07941-N6	Post Digestion Spike

* Homogenization Test: ICP & GEA

** Fusion Radchem: Total Alpha, Total Beta, Sr-90, PU-239/240, GEA, U)

CORE 99 SEGMENT 2 ANALYSES

Analysis	Segment 2 Drainable Liquid	Quarter Segment 2A Unhomogenized	Quarter Segment 2D Unhomogenized	Quarter Segment 2A	Quarter Segment 2D	
TGA	95-07932	95-07933	95-07934			Run in Duplicate
Acid Digestion	95-07932-A1					Acid Digestion Sample (ICP, Radchem *)
	95-07932-A2					Acid Digestion Duplicate
	95-07932-A3					Methods Blank (one per batch)
	95-07932-A4					Matrix Spike (one per batch)
	95-07932-A5					Blank Spike (one per batch)
Water Leach	95-07932-C1			95-07942-C1	95-07943-C1	Water Leach Sample **
	95-07932-C2			95-07942-C2	95-07943-C2	Water Leach Duplicate
	95-07932-C3			95-07942-C3	95-07943-C3	Methods Blank (one per batch)
	95-07932-C4			95-07942-C4	95-07943-C4	Spike (IC Only) (one per batch)
	95-07932-C5			95-07942-C5	95-07943-C5	Blank Spike (IC Only) (one per batch)
Direct	95-07932-D1			95-07942-D1	95-07943-D1	Direct Sample ***
	95-07932-D2			95-07942-D2	95-07943-D2	Direct Duplicate
	95-07932-D3			95-07942-D3	95-07943-D3	Methods Blank (one per batch)
Total Cyanide	95-07932-G1			95-07942-G1	95-07943-G1	Total CN Sample
	95-07932-G2			95-07942-G2	95-07943-G2	Total CN Duplicate
	95-07932-G3			95-07942-G3	95-07943-G3	Methods Blank (one per batch)
	95-07932-G4			95-07942-G4	95-07943-G4	Matrix Spike (one per batch)
	95-07932-G5			95-07942-G5	95-07943-G5	Blank Spike (one per batch)
Fusion Dissolution KOH				95-07942-H1	95-07943-H1	KOH Fusion Sample (ICP, Radchem****)
				95-07942-H2	95-07943-H2	KOH Fusion Duplicate
				95-07942-H3	95-07943-H3	Methods Blank (one per batch)
				95-07942-H5	95-07943-H5	LCS (one per prep)
				95-07942-H6	95-07943-H6	Post Digestion Spike (ICP Only)
Carbon	95-07932-J1			95-07942-J1	95-07943-J1	Carbon Analysis Sample (TIC/TOC/TC)
	95-07932-J2			95-07942-J2	95-07943-J2	Carbon Analysis Duplicate
	95-07932-J3			95-07942-J3	95-07943-J3	Methods Blank (one per batch)
	95-07932-J4			95-07942-J4	95-07943-J4	Matrix Spike (one per batch)
Fusion Dissolution Na2O2				95-07942-N1	95-07943-N1	Na2O2 Fusion Sample (ICP)
				95-07942-N2	95-07943-N2	Na2O2 Fusion Duplicate (one per batch)
				95-07942-N3	95-07943-N3	Methods Blank (one per batch)
				95-07942-N5	95-07943-N5	LCS (one per prep)
				95-07942-N6	95-07943-N6	Post Digestion Spike

* Acid Digestion Radchem: Total Alpha, Total Beta, GEA
** Water Leach: IC on Drainable Liquid and Quarter Segments, , ICP on Quarter Segments
*** Direct Sample: DSC on Drainable Liquid, DSC and Density on Quarter Segments
**** Fusion Radchem: Total Alpha, Total Beta, Sr-90, PU-239/240, GEA, U)

CORE 99 SEGMENT 3 ANALYSES

Analysis	Segment 3 Drainable Liquid	Quarter Segment 3A Unhomogenized	Quarter Segment 3A	
TGA	95-07935	95-07936		Run in Duplicate
Acid Digestion	95-07935-A1			Acid Digestion Sample (ICP, Radchem *)
	95-07935-A2			Acid Digestion Duplicate
	95-07935-A3			Methods Blank (one per batch)
	95-07935-A4			Spike (one per batch)
	95-07935-A5			Blank Spike (one per batch)
Water Leach	95-07935-C1		95-07944-C1	Water Leach Sample **
	95-07935-C2		95-07944-C2	Water Leach Duplicate
	95-07935-C3		95-07944-C3	Methods Blank (one per batch)
	95-07935-C4		95-07944-C4	Spike (IC Only) (one per batch)
	95-07935-C5		95-07944-C5	Blank Spike (IC Only) (one per batch)
Direct	95-07935-D1		95-07944-D1	Direct Sample ***
	95-07935-D2		95-07944-D2	Direct Duplicate
	95-07935-D3		95-07944-D3	Methods Blank (one per batch)
Total Cyanide	95-07935-G1		95-07944-G1	Total CN Sample
	95-07935-G2		95-07944-G2	Total CN Duplicate
	95-07935-G3		95-07944-G3	Methods Blank (one per batch)
	95-07935-G4		95-07944-G4	Matrix Spike (one per batch)
	95-07935-G5		95-07944-G5	Blank Spike (one per batch)
Fusion Dissolution KOH			95-07944-H1	KOH Fusion Sample (ICP, Radchem****)
			95-07944-H2	KOH Fusion Duplicate
			95-07944-H3	Methods Blank (one per batch)
			95-07944-H5	LCS (one per prep)
			95-07944-H6	Post Digestion Spike (ICP Only)
Carbon	95-07935-J1		95-07944-J1	Carbon Analysis Sample (TIC/TOC/TC)
	95-07935-J2		95-07944-J2	Carbon Analysis Duplicate
	95-07935-J3		95-07944-J3	Methods Blank (one per batch)
	95-07935-J4		95-07944-J4	Matrix Spike (one per batch)
Fusion Dissolution Na2O2			95-07944-N1	Na2O2 Fusion Sample (ICP)
			95-07944-N2	Na2O2 Fusion Duplicate (one per batch)
			95-07944-N3	Methods Blank (one per batch)
			95-07944-N5	LCS (one per prep)
			95-07944-N6	Post Digestion Spike

* Acid Digestion Radchem: Total Alpha, Total Beta, GEA
** Water Leach: IC on Drainable Liquid and Quarter Segment, ICP on Quarter Segment
*** Direct Samples: DSC on Drainable Liquid, DSC and Density on Quarter Segment
**** Fusion Radchem: Total Alpha, Total Beta, Sr-90, PU-239/240, GEA, U)

CORE 99 SEGMENT 4 ANALYSES

Analysis	Quarter Segment 4A Unhomogenized	Quarter Segment 4B Unhomogenized	Quarter Segment 4C Unhomogenized	Quarter Segment 4D Unhomogenized	Quarter Segment 4A	Quarter Segment 4B	Quarter Segment 4C	Quarter Segment 4D	
TGA	95-07937	95-07938	95-07939	95-07940					Run in Duplicate
Water Leach					95-07945-C1	95-07946-C1	95-07947-C1	95-07948-C1	Water Leach Sample (IC, ICP)
					95-07945-C2	95-07946-C2	95-07947-C2	95-07948-C2	Water Leach Duplicate
					95-07945-C3	95-07946-C3	95-07947-C3	95-07948-C3	Methods Blank (one per batch)
					95-07945-C4	95-07946-C4	95-07947-C4	95-07948-C4	Spike (IC Only) (one per batch)
					95-07945-C5	95-07946-C5	95-07947-C5	95-07948-C5	Blank Spike (IC Only) (one per batch)
Direct					95-07945-D1	95-07946-D1	95-07947-D1	95-07948-D1	DSC & Density Sample
					95-07945-D2	95-07946-D2	95-07947-D2	95-07948-D2	DSC & Density Duplicate
					95-07945-D3	95-07946-D3	95-07947-D3	95-07948-D3	Methods Blank (one per batch)
Total Cyanide					95-07945-G1	95-07946-G1	95-07947-G1	95-07948-G1	Total CN Sample
					95-07945-G2	95-07946-G2	95-07947-G2	95-07948-G2	Total CN Duplicate
					95-07945-G3	95-07946-G3	95-07947-G3	95-07948-G3	Methods Blank (one per batch)
					95-07945-G4	95-07946-G4	95-07947-G4	95-07948-G4	Matrix Spike (one per batch)
					95-07945-G5	95-07946-G5	95-07947-G5	95-07948-G5	Blank Spike (one per batch)
Homogenization Test Fusion Dissolution KOH						95-07946-H1T			Homogenization Test Fusion Sample - Top *
						95-07946-H2T			Homogenization Test Fusion Duplicate - Top
						95-07946-HCB			Homogenization Test Fusion Blank
						95-07946-H1B			Homogenization Test Fusion Sample - Bottom
						95-07946-H2B			Homogenization Test Fusion Duplicate - Bottom
Fusion Dissolution KOH					95-07945-H1	95-07946-H1	95-07947-H1	95-07948-H1	KOH Fusion Sample (ICP, Radchem**)
					95-07945-H2	95-07946-H2	95-07947-H2	95-07948-H2	KOH Fusion Duplicate
					95-07945-H3	95-07946-H3	95-07947-H3	95-07948-H3	Methods Blank (one per batch)
					95-07945-H5	95-07946-H5	95-07947-H5	95-07948-H5	LCS (one per prep)
					95-07945-H6	95-07946-H6	95-07947-H6	95-07948-H6	Post Digestion Spike (ICP Only)
Carbon					95-07945-J1	95-07946-J1	95-07947-J1	95-07948-J1	Carbon Analysis Sample (TIC/TOC/TC)
					95-07945-J2	95-07946-J2	95-07947-J2	95-07948-J2	Carbon Analysis Duplicate
					95-07945-J3	95-07946-J3	95-07947-J3	95-07948-J3	Methods Blank (one per batch)
					95-07945-J4	95-07946-J4	95-07947-J4	95-07948-J4	Matrix Spike (one per batch)
Fusion Dissolution Na2O2					95-07945-N1	95-07946-N1	95-07947-N1	95-07948-N1	Na2O2 Fusion Sample (ICP)
					95-07945-N2	95-07946-N2	95-07947-N2	95-07948-N2	Na2O2 Fusion Duplicate (one per batch)
					95-07945-N3	95-07946-N3	95-07947-N3	95-07948-N3	Methods Blank (one per batch)
					95-07945-N5	95-07946-N5	95-07947-N5	95-07948-N5	LCS (one per prep)
					95-07945-N6	95-07946-N6	95-07947-N6	95-07948-N6	Post Digestion Spike

* Homogenization Test: ICP & GEA
** Fusion Radchem: Total Alpha, Total Beta, Sr-90, PU-239/240, GEA, U)

TANK CHARACTERIZATION
TEST INSTRUCTIONS FOR ANALYSES OF
SST 241-BY-108, CORE 99 SEGMENTS 1-4

DATE PREPARED: September 6, 1995 PREPARED BY: KL Silvers

SAMPLE NUMBERS: 95-07931 - 95-07948

APPROVED BY: [signature] DATE: 9/6/95

CONTROLLING DOCUMENTS: Project No: 21372

WHC TCP: Tank 241-BY-108 Tank Characterization Plan (WHC-SD-WM-TP-275)
Revision OE - ECN No: 6621348 (August, 1995)
PNL QAP: MCS-033, Revision 2 (April, 1995)
Controlling Procedure: PNL-ALO-010

INTRODUCTION

This Test Instruction (TI) defines the scope of work to be completed on Single-Shell Tank (SST) 241-BY-108, Core 99 Segments 1, 2, 3 and 4. The samples from this tank will be analyzed according to TCP (WHC-SD-WM-TP-275 Rev. OE).

This TI contains instructions for the preparation and analyses by the ACL of all segments, quarter-segments, and drainable liquids from Core 99.

All activities identified in this Test Instruction shall be charged against Project 21372. All analyses are to be completed at Impact Level 2. All analyses are to be completed following the identified procedures. Any deviations to the procedures must be documented and this documentation must accompany the analytical data. All analytical data are returned to the Laboratory Support Office.

SAL SAMPLE PREPARATION INSTRUCTIONS FOR SEGMENTS AND QUARTER-SEGMENTS:

The following tank material samples were transferred from the HLRF:

CORE IDENTIFICATION	ACL NUMBER
Core 99 - Segment 1 (homogenized, mechanical)	95-07941
Core 99 - Segment 2 (drainable liquid)	95-07932
Core 99 - Quarter-Segment 2A (homogenized, manual)	95-07942
Core 99 - Quarter-Segment 2D (homogenized, manual)	95-07943
Core 99 - Segment 3 (drainable liquid)	95-07935
Core 99 - Quarter-Segment 3A (homogenized, manual)	95-07944

CORE IDENTIFICATION	ACL NUMBER
Core 99 - Quarter-Segment 4A (homogenized, mechanical)	95-07945
Core 99 - Quarter-Segment 4B (homogenized, mechanical)	95-07946
Core 99 - Quarter-Segment 4C (homogenized, mechanical)	95-07947
Core 99 - Quarter-Segment 4D (homogenized, mechanical)	95-07948

The following sample preparation steps are required:

Method		Procedure
Acid Digestion for ICP - liquid (QC Requirements: Duplicate, Blank, Spike, Spike Control)	(WPkg K28862)	PNL-ALO-128
Caustic Fusion - NaOH (QC Requirements - Radchem: Duplicate, Blank) (QC Requirements - ICP : 1 Duplicate per batch, Blank, Post Digestion Spike)	(WPkg K28863)	PNL-ALO-114
Caustic Fusion - KOH (QC Requirements: Duplicate, Blank, Post Digestion Spike)	(WPkg K28863)	PNL-ALO-115
Water Leach (QC Requirements - IC: Duplicate, Blank, Spike, Spike Control) (QC Requirements - ICP: Duplicate, Blank)	(WPkg K28864)	PNL-ALO-103
Total CN Prep (QC Requirements: Duplicate, Blank, Spike, Spike Control)	(WPkg K28868)	PNL-ALO-285

REQUESTED ANALYSES FOR SEGMENTS & QUARTER-SEGMENTS:

Requested Analysis	Procedure Number	Task Leader	WP#
ICP (Acid, Fusion, Water Leach)	PNL-ALO-211	Urie	K28866
IC (Water Leach)	PNL-ALO-212	Urie	K28867
CN (Direct)	PNL-ALO-285	Urie	K28868
TOC by Hot Persulfate (Direct)	PNL-ALO-381	Steele	K28869
Total Alpha (Fusion)	PNL-ALO-421	Greenwood	K28870
AEA (Pu-239/240) (Fusion)	PNL-ALO-422/423	Greenwood	K28871
GEA (Fusion)	PNL-ALO-450	Greenwood	K28872

Requested Analysis	Procedure Number	Task Leader	WP#
Total Beta (Fusion)	PNL-ALO-430/431	Greenwood	K28873
Sr-90 (Fusion)	PNL-ALO-433/431	Greenwood	K28874
U-Laser (Fusion)	PNL-ALO-445	Greenwood	K28875

SAMPLE IDENTIFICATION SYSTEM FOR SEGMENT AND QUARTER-SEGMENTS:

The following sample identification system will be followed. All samples will be idenitified as 95-XXXXX-Y#, where 95-XXXXX is the ACL number. The "Y" letter will identify a Sample Preparation Method. The "#" identifies replicate analyses using the sample preparation method (i.e., duplicates, spikes, blanks, etc.). The Sample Preparation Methods codes are:

A	Acid Digestion for ICP
C	Water Leach
D	Direct
G	Cyanide Sample Prep
H	Caustic Fusion - KOH
J	Carbon
N	Caustic Fusion - NaOH

CORE 99 SEGMENT 1 ANALYSES

Analysis	Segment 1 Unhomogenized	Segment 1	
TGA	95-07931		Run in Duplicate
Water Leach		95-07941-C1	Water Leach Sample (IC, ICP)
		95-07941-C2	Water Leach Duplicate
		95-07941-C3	Methods Blank
		95-07941-C4	Spike (IC Only)
		95-07941-C5	Spike Control (IC Only)
Direct		95-07941-D1	DSC & Density Sample
		95-07941-D2	DSC & Density Duplicate
		95-07941-D3	Methods Blank
Total Cyanide		95-07941-G1	Total CN Sample
		95-07941-G2	Total CN Duplicate
		95-07941-G3	Methods Blank (min 1 per batch)
		95-07941-G4	Matrix Spike
		95-07941-G5	Spike Control
Homogenization Test Fusion Dissolution KOH		95-07941-H1T	Homogenization Test Fusion Sample - Top *
		95-07941-H2T	Homogenization Test Fusion Duplicate - Top
		95-07941-HCB	Homogenization Test Fusion Blank
		95-07941-H1B	Homogenization Test Fusion Sample - Bottom
		95-07941-H2B	Homogenization Test Fusion Duplicate - Bottom
Fusion Dissolution KOH		95-07941-H1	KOH Fusion Sample (ICP, Radchem**)
		95-07941-H2	KOH Fusion Duplicate
		95-07941-H3	Methods Blank
		95-07941-H6	Post Digestion Spike (ICP Only)
Carbon		95-07941-J1	Carbon Analysis Sample (TIC/TOC/TC)
		95-07941-J2	Carbon Analysis Duplicate
		95-07941-J3	Methods Blank
		95-07941-J4	Matrix Spike
Fusion Dissolution NaOH		95-07941-N1	NaOH Fusion Sample (ICP)
		95-07941-N2	NaOH Fusion Duplicate (one per batch)
		95-07941-N3	Methods Blank
		95-07941-N6	Post Digestion Spike

* Homogenization Test: ICP & GEA
** Fusion Radchem: Total Alpha, Total Beta, Sr-90, PU-239/240, GEA, U)

CORE 99 SEGMENT 2 ANALYSES

Analysis	Segment 2 Drainable Liquid	Quarter Segment 2A Unhomogenized	Quarter Segment 2D Unhomogenized	Quarter Segment 2A	Quarter Segment 2D	
TGA	95-07932	95-07933	95-07934			Run in Duplicate
Acid Digestion	95-07932-A1					Acid Digestion Sample (ICP)
	95-07932-A2					Acid Digestion Duplicate
	95-07932-A3					Methods Blank (min 1 per batch)
	95-07932-A4					Matrix Spike
	95-07932-A5					Spike Control
Water Leach	95-07932-C1			95-07942-C1	95-07943-C1	Water Leach Sample *
	95-07932-C2			95-07942-C2	95-07943-C2	Water Leach Duplicate
	95-07932-C3			95-07942-C3	95-07943-C3	Methods Blank
	95-07932-C4			95-07942-C4	95-07943-C4	Spike (IC Only)
	95-07932-C5			95-07942-C5	95-07943-C5	Spike Control (IC Only)
Direct	95-07932-D1			95-07942-D1	95-07943-D1	Direct Sample **
	95-07932-D2			95-07942-D2	95-07943-D2	Direct Duplicate
	95-07932-D3			95-07942-D3	95-07943-D3	Methods Blank
Total Cyanide	95-07932-G1			95-07942-G1	95-07943-G1	Total CN Sample
	95-07932-G2			95-07942-G2	95-07943-G2	Total CN Duplicate
	95-07932-G3			95-07942-G3	95-07943-G3	Methods Blank (min 1 per batch)
	95-07932-G4			95-07942-G4	95-07943-G4	Matrix Spike
	95-07932-G5			95-07942-G5	95-07943-G5	Spike Control
Fusion Dissolution KOH				95-07942-H1	95-07943-H1	KOH Fusion Sample (ICP, Radchem***)
				95-07942-H2	95-07943-H2	KOH Fusion Duplicate
				95-07942-H3	95-07943-H3	Methods Blank
				95-07942-H6	95-07943-H6	Post Digestion Spike (ICP Only)
Carbon	95-07932-J1			95-07942-J1	95-07943-J1	Carbon Analysis Sample (TIC/TOC/TC)
	95-07932-J2			95-07942-J2	95-07943-J2	Carbon Analysis Duplicate
	95-07932-J3			95-07942-J3	95-07943-J3	Methods Blank
	95-07932-J4			95-07942-J4	95-07943-J4	Matrix Spike
Fusion Dissolution NaOH				95-07942-N1	95-07943-N1	NaOH Fusion Sample (ICP)
				95-07942-N2	95-07943-N2	NaOH Fusion Duplicate (one per batch)
				95-07942-N3	95-07943-N3	Methods Blank
				95-07942-N6	95-07943-N6	Post Digestion Spike

* Water Leach: IC on Drainable Liquid and Quarter Segments, , ICP on Quarter Segments
** Direct Sample: DSC on Drainable Liquid, DSC and Density on Quarter Segments
*** Fusion Radchem: Total Alpha, Total Beta, Sr-90, PU-239/240, GEA, U)

CORE 99 SEGMENT 3 ANALYSES

Analysis	Segment 3 Drainable Liquid	Quarter Segment 3A Unhomogenized	Quarter Segment 3A	
TGA	95-07935	95-07936		Run in Duplicate
Acid Digestion	95-07935-A1			Acid Digestion Sample (ICP)
	95-07935-A2			Acid Digestion Duplicate
	95-07935-A3			Methods Blank
	95-07935-A4			Spike
	95-07935-A5			Spike Control
Water Leach	95-07935-C1		95-07944-C1	Water Leach Sample *
	95-07935-C2		95-07944-C2	Water Leach Duplicate
	95-07935-C3		95-07944-C3	Methods Blank
	95-07935-C4		95-07944-C4	Spike (IC Only)
	95-07935-C5		95-07944-C5	Spike Control (IC Only)
Direct	95-07935-D1		95-07944-D1	Direct Sample **
	95-07935-D2		95-07944-D2	Direct Duplicate
	95-07935-D3		95-07944-D3	Methods Blank
Total Cyanide	95-07935-G1		95-07944-G1	Total CN Sample
	95-07935-G2		95-07944-G2	Total CN Duplicate
	95-07935-G3		95-07944-G3	Methods Blank (min 1 per batch)
	95-07935-G4		95-07944-G4	Matrix Spike
	95-07935-G5		95-07944-G5	Spike Control
Fusion Dissolution KOH			95-07944-H1	KOH Fusion Sample (ICP, Radchem***)
			95-07944-H2	KOH Fusion Duplicate
			95-07944-H3	Methods Blank
			95-07944-H6	Post Digestion Spike (ICP Only)
Carbon	95-07935-J1		95-07944-J1	Carbon Analysis Sample (TIC/TOC/TC)
	95-07935-J2		95-07944-J2	Carbon Analysis Duplicate
	95-07935-J3		95-07944-J3	Methods Blank
	95-07935-J4		95-07944-J4	Matrix Spike
Fusion Dissolution NaOH			95-07944-N1	NaOH Fusion Sample (ICP)
			95-07944-N2	NaOH Fusion Duplicate (one per batch)
			95-07944-N3	Methods Blank
			95-07944-N6	Post Digestion Spike

* Water Leach: IC on Drainable Liquid and Quarter Segment, ICP on Quarter Segment
** Direct Samples: DSC on Drainable Liquid, DSC and Density on Quarter Segment
*** Fusion Radchem: Total Alpha, Total Beta, Sr-90, PU-239/240, GEA, U)

CORE 99 SEGMENT 4 ANALYSES

Analysis	Quarter Segment 4A Unhomogenized	Quarter Segment 4B Unhomogenized	Quarter Segment 4C Unhomogenized	Quarter Segment 4D Unhomogenized	Quarter Segment 4A	Quarter Segment 4B	Quarter Segment 4C	Quarter Segment 4D	
TGA	95-07937	95-07938	95-07939	95-07940					Run In Duplicate
Water Leach					95-07945-C1	95-07946-C1	95-07947-C1	95-07948-C1	Water Leach Sample (IC, ICP)
					95-07945-C2	95-07946-C2	95-07947-C2	95-07948-C2	Water Leach Duplicate
					95-07945-C3	95-07946-C3	95-07947-C3	95-07948-C3	Methods Blank
					95-07945-C4	95-07946-C4	95-07947-C4	95-07948-C4	Spike (IC Only)
					95-07945-C5	95-07946-C5	95-07947-C5	95-07948-C5	Spike Control (IC Only)
Direct					95-07945-D1	95-07946-D1	95-07947-D1	95-07948-D1	DSC & Density Sample
					95-07945-D2	95-07946-D2	95-07947-D2	95-07948-D2	DSC & Density Duplicate
					95-07945-D3	95-07946-D3	95-07947-D3	95-07948-D3	Methods Blank
Total Cyanide					95-07945-G1	95-07946-G1	95-07947-G1	95-07948-G1	Total CN Sample
					95-07945-G2	95-07946-G2	95-07947-G2	95-07948-G2	Total CN Duplicate
					95-07945-G3	95-07946-G3	95-07947-G3	95-07948-G3	Methods Blank (min 1 per batch)
					95-07945-G4	95-07946-G4	95-07947-G4	95-07948-G4	Matrix Spike
					95-07945-G5	95-07946-G5	95-07947-G5	95-07948-G5	Spike Control
Homogenization Test Fusion Dissolution KOH						95-07946-H1T			Homogenization Test Fusion Sample - Top *
						95-07946-H2T			Homogenization Test Fusion Duplicate - Top
						95-07946-HCB			Homogenization Test Fusion Blank
						95-07946-H1B			Homogenization Test Fusion Sample - Bottom
						95-07946-H2B			Homogenization Test Fusion Duplicate - Bottom
Fusion Dissolution KOH					95-07945-H1	95-07946-H1	95-07947-H1	95-07948-H1	KOH Fusion Sample (ICP, Radchem**)
					95-07945-H2	95-07946-H2	95-07947-H2	95-07948-H2	KOH Fusion Duplicate
					95-07945-H3	95-07946-H3	95-07947-H3	95-07948-H3	Methods Blank
					95-07945-H6	95-07946-H6	95-07947-H6	95-07948-H6	Post Digestion Spike (ICP Only)
Carbon					95-07945-J1	95-07946-J1	95-07947-J1	95-07948-J1	Carbon Analysis Sample (TIC/TOC/TC)
					95-07945-J2	95-07946-J2	95-07947-J2	95-07948-J2	Carbon Analysis Duplicate
					95-07945-J3	95-07946-J3	95-07947-J3	95-07948-J3	Methods Blank
					95-07945-J4	95-07946-J4	95-07947-J4	95-07948-J4	Matrix Spike
Fusion Dissolution NaOH					95-07945-N1	95-07946-N1	95-07947-N1	95-07948-N1	NaOH Fusion Sample (ICP, Radchem**)
					95-07945-N2	95-07946-N2	95-07947-N2	95-07948-N2	NaOH Fusion Duplicate (one per batch)
					95-07945-N3	95-07946-N3	95-07947-N3	95-07948-N3	Methods Blank
					95-07945-N6	95-07946-N6	95-07947-N6	95-07948-N6	Post Digestion Spike

* Homogenization Test: ICP & GEA
** Fusion Radchem: Total Alpha, Total Beta, Sr-90, PU-239/240, GEA, U)

A5 - SAMPLE PREP DATA SHEETS

DON'T SAY IT -- *Write It!* **Date:** September 20, 1995

To: Memo To File/BY108 PM/QAQC Mgr **From:** Rick Steele

Subject: Tank BY-108 Sample Prep Documentational Error

The "Controlling Procedure" block on many Sample Prep Sheets supporting BY-108 characterization incorrectly listed "PNL-ALO-110". The correct procedural citation should have been "PNL-ALO-010" in all cases. The major effect of PNL-ALO-010 is that it invokes internal Chain of Custody. This was accomplished real time as samples traversed through the laboratory.

Please address any questions or concerns related to this error directly to me.

E54-3000-101 (10/89)

Battelle
Pacific Northwest Laboratories

Project Number 21372

Internal Distribution

JM Latkovich
KL Silvers
OP Bredt
KJ Smith
File/LB

Date September 19, 1995

To TWRS Support Project File

From KJ Kuhl-Klinger

Subject Observations Performed on BY108

This memorandum serves as confirmation that all sample preparation activities (i.e., Water Leach, Acid Extraction and Fusion) used to process BY108 samples for analyses were subject to continual observation. The observations were conducted between 9/6/95 and 9/11/95. Observations were not conducted during homogeneity testing nor were they conducted on subsequent analyses such as TOC, CN and Hg.

The observations were conducted as assistance to the SAL but were also conducted to gain better understanding of the complexities associated with this scope of work.

Observations were conducted by one or more of the following personnel:

Kristine Kuhl-Klinger, Quality Operations — 9/19/95 Date

Ofelia Bredt, Quality Operations — 9/20/95 Date

Karla Smith, SAL Operations — 9/19/95 Date

E54-1900-001 (10/89)

KOH FUSION AND SOLUBILITY TEST FOR HOMOGENIZATION CHECK

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DATE TO QC: 09/19/95

DATA QUALITY REVIEW

I have reviewed the following data for completeness and for compliance with project requirements.

Analyte - Fusion and Solubility Test for Homogenization Checks

Data Package/Report - BY-108 Core 99

Project No. - 21372

ACL Numbers -	95-07941-H1T	95-07941-H2T	95-07941-HCB	95-07941-H1B
	95-07941-H2B			
	95-07946-H1T	95-07946-H2T	95-07946-HCB	95-07946-H1B
	95-07946-H2B			

Julia Buck

PNL/ACL Quality Representative

9/20/95

Date

Battelle Pacific Northwest Laboratories
Analytical Chemistry Laboratory
Shielded Analytical Laboratory

Shielded Analytical Laboratory FUSIONS

Tank BY-108 Core(s) 99

Project Id: TWC/21372 WP Number: K28863

Test Instruction: TI95-TWC-02 Procedure: PNL-ALO-115

	Sample Ident.	Wet Sample Gross Wt (g)	Wet Sample Tare Wt (g)	Wet Sample Net Wt (g)	Dry Sample Gross Wt (g)	Dry Sample Tare Wt (g)	Dry Sample Net Wt (g)
1	95-07946-H1T	44.1871	44.0869	0.1002			
2	95-07946-H2T	45.8278	45.7286	0.0992			
Blk 3	95-07946-HCB	~~45.7378~~	45.6777				
4	95-07946-H1B	45.7378	45.6262	0.1116			
5	95-07946-H2B	45.5056	45.4099	0.0957			

All samples brought to 100 mL total volume,

4.9400 $\bar{x}$ 4.9493 ~~see~~ 9/1/95

4.9544 s 0.0082

4.9434 RSD 17%

4.9485

4.9603 Pef. 4.9653 ml

M&TE:

✓ Mettler AE160 (360-06-01-016) ___ Mettler AE200 (362-06-01-038)

___ Sartorius R200D (360-06-01-024)

Analyst: [signature] Date: 9-1-95 Reviewer: [signature] Date: 9/1/95

Battelle Pacific Northwest Laboratories
Analytical Chemistry Laboratory
Shielded Analytical Laboratory

Shielded Analytical Laboratory
FUSIONS

Tank BY-108 Core(s) 99

Project Id: TWC/21372 WP Number: K28863

Test Instruction: TI95-TWC-02 Procedure: PNL-ALO-115

	Sample Ident.	Wet Sample Gross Wt (g)	Wet Sample Tare Wt (g)	Wet Sample Net Wt (g)	Dry Sample Gross Wt (g)	Dry Sample Tare Wt (g)	Dry Sample Net Wt (g)
6	95-07941-H1T	46.4294	46.3289	0.1005			
7	95-07941-H2T	44.8966	44.7940	0.1026			
Blk 8	95-07941-HCB	~~XXXX~~	45.8179	~~0.1028~~ RTS 9/1/95			
9	95-07941-H1B	52.8967	52.7939	0.1028			
10	95-07941-H2B	~~44.5022~~ 39.9403	~~44.4021~~ 39.8114	0.1289			

All samples brought to 100 ml total volume.
See 95-07946 ~~Fe~~ Fusion Bench Sheet For Pipet Info.

M&TE:

✓ Mettler AE160 (360-06-01-016) ___ Mettler AE200 (362-06-01-038)

___ Sartorius R200D (360-06-01-024)

Analyst: [signature] Date: 9-1-95 Reviewer: [signature] Date: 9/1/95

Battelle Pacific Northwest Laboratories
Analytical Chemistry Laboratory
325 Shielded Analytical Laboratory

SAMPLE PREP SHEET
(325 SHIELDED ANALYTICAL LABORATORY)

TI NO.: TI95-TWC-02 PROJECT NO.: 21372 WBS NO.: 020501 SAMPLE TYPE: SLUDGE

ISSUED BY: Rick T. Steele DATE: 8/28/95 PREP TYPE: NI/KOH FUSION

ANALYST: Clyde Chamberlain DATE: 9-1-95 CONTROLLING PROCEDURE: PNL-ALO-010

REVIEW: Rick T. Steele D TE: 9/1/95 QA PLAN: MCS-033 IMPACT LEVEL: II

CORE ID: 99 TANK ID: BY-108

WORK PACKAGE NUMBER	ALO NUMBER	ANALYTICAL PROCEDURE	ANALYTE OR ANALYSIS	SAMPLE WT	WATER WT (g)	TOTAL VOL (mL)	SPIKE ID	SPIKE VOL (mL)	DILUTION FACTOR	DILUTION MATRIX	PIPET CALIB (mL)	MISC
K28872	95-07941-H1T	PNL-ALO-450	GEA	0.1005		100					4.9653	SAMP
	95-07941-H2T			0.1026								SAMP DUP
	95-07941-HCB											BLANK
	95-07941-H1B			0.1028								SAMP
	95-07941-H2B			0.1289								SAMP DUP

Delivered 9.9305 ml of each sample to the lab.

PAGE 1 OF 1

WHC-SD-WM-DP-145, REV. 1

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Battelle Pacific Northwest Laboratories
Analytical Chemistry Laboratory
325 Shielded Analytical Laboratory

SAMPLE PREP SHEET
(325 SHIELDED ANALYTICAL LABORATORY)

TI NO.: TI95-TWC-02 PROJECT NO.: 21372 WBS NO.: 020501 SAMPLE TYPE: SLUDGE

ISSUED BY: Rick T. Steele DATE: 8/28/95 PREP TYPE: Ni/KOH FUSION

ANALYST: [signature] DATE: 9-1-95 CONTROLLING PROCEDURE: PNL-ALO-010

REVIEW: [signature] D TE: 9/1/95 QA PLAN: MCS-033 IMPACT LEVEL: II

CORE ID: 99 TANK ID: BY-108

WHC-SD-WM-DP-165, REV. 1

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WORK PACKAGE NUMBER	ALO NUMBER	ANALYTICAL PROCEDURE	ANALYTE OR ANALYSIS	SAMPLE WT	WATER WT (g)	TOTAL VOL (mL)	SPIKE ID	SPIKE VOL (mL)	DILUTION FACTOR	DILUTION MATRIX	PIPET CALIB (mL)	MISC
K28872	95-07946-H1T	PNL-ALO-450	GEA	0.1002		100					4.9653	SAMP
	95-07946-H2T			0.0992								SAMP DUP
	95-07946-HCB											BLANK
	95-07946-H1B			0.1116								SAMP
	95-07946-H2B			0.0957								SAMP DUP

Delivered 9.9305ml of each sample to the lab.

Battelle Pacific Northwest Laboratories
Analytical Chemistry Laboratory
325 Shielded Analytical Laboratory

SAMPLE PREP SHEET
(325 SHIELDED ANALYTICAL LABORATORY)

TI NO.: TI95-TWC-02 PROJECT NO.: 21372 WBS NO.: 020501 SAMPLE TYPE: SLUDGE

ISSUED BY: Rick T. Steele DATE: 8/28/95 PREP TYPE: NI/KOH FUSION

ANALYST: Clyde Chamberlain DATE: 9-1-95 CONTROLLING PROCEDURE: PNL-ALO-010

REVIEW: Rick T. Steele D TE: 9/1/95 QA PLAN: MCS-033 IMPACT LEVEL: II

CORE ID: 99 TANK ID: BY-108

WORK PACKAGE NUMBER	ALO NUMBER	ANALYTICAL PROCEDURE	ANALYTE OR ANALYSIS	SAMPLE WT	WATER WT (g)	TOTAL VOL (mL)	SPIKE ID	SPIKE VOL (mL)	DILUTION FACTOR	DILUTION MATRIX	PIPET CALIB (mL)	MISC
K28866	95-07941-H1T	PNL-ALO-211	ICP	0.1005		100						SAMP
	95-07941-H2T			0.1026								SAMP DUP
	95-07941-HCB											BLANK
	95-07941-H1B			0.1028								SAMP
	95-07941-H2B			0.1289								SAMP DUP

PAGE 1 OF 1

Battelle Pacific Northwest Laboratories
Analytical Chemistry Laboratory
325 Shielded Analytical Laboratory

SAMPLE PREP SHEET
(325 SHIELDED ANALYTICAL LABORATORY)

TI NO.: TI95-TWC-02 PROJECT NO.: 21372 WBS NO.: 020501 SAMPLE TYPE: SLUDGE

ISSUED BY: Rick T. Steele DATE: 8/28/95 PREP TYPE: NI/KOH FUSION

ANALYST: [signature] DATE: 9-1-95 CONTROLLING PROCEDURE: PNL-ALO-010

REVIEW: [signature] DATE: 9/1/95 QA PLAN: MCS-033 IMPACT LEVEL: II

CORE ID: 99 TANK ID: BY-108

WORK PACKAGE NUMBER	ALO NUMBER	ANALYTICAL PROCEDURE	ANALYTE OR ANALYSIS	SAMPLE WT	WATER WT (g)	TOTAL VOL (mL)	SPIKE ID	SPIKE VOL (mL)	DILUTION FACTOR	DILUTION MATRIX	PIPET CALIB (mL)	MISC
K28866	95-07946-H1T	PNL-ALO-211	ICP	0.1002		100						SAMP
	95-07946-H2T			0.0992								SAMP DUP
	95-07946-HCB											BLANK
	95-07946-H1B			0.1116								SAMP
	95-07946-H2B			0.0957								SAMP DUP

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WHC-SD-WM-DP-145, REV. 1

Battelle Pacific Northwest Laboratories
Analytical Chemistry Laboratory
Shielded Analytical Laboratory/Sample Receiving Laboratory

PNL-ALO-129 — HNO_3-HCl Acid Extraction of Solids for Metals Analysis Using a Dry-Block Heater

Client name: Kurt Silvers
Work Auth. Doc (WAD): TI95-TWC-02 / ASR 2386
Tank/Core/Project: Tank BY-108, Core 99, Segment 1 and Qtr Segment 4B / Tank Waste Characterization
Special instructions

Work package number: K28866
Project number: 21372
PNL QA plan: MCS-033
PNL impact level: II
Prep. lab (SAL/SRPL/other): SAL
Preparation batch number: N/A

	ACL Sample ID	ACL order number or Client sample ID	Vial identifier	Vial weight (g)	Vial & sample weight (g)	Sample weight (g)	Spike added Vol. (ml)	Spike added Weight. (g)	Final solution Volume (ml)	Process Factor (1)
1	95-07941-H1T	Segment 1 Top		21.5632	22.0188	0.4556			25	
2	95-07941-H2T	Segment 1 Top-Dup		21.6672	22.1335	0.4663				
3	95-07941-HCB	Methods Blank								
4	95-07946-H1T	Qtr Segment 4B Top		21.5403	22.0056	0.4653				
5	95-07946-H2T	Qtr Segment 4B Top-Dup		21.4616	22.0536	0.5920				
6										
7										
8										
9										
10										
11										
12										
13										
14										

Analyst's sample preparation comments:
11.01X dilutions of All samples Above were made prior to distribution.

Dil. Factor: .9980 → $\frac{9.9903}{.9980}$ = $\boxed{11.01X}$

Spike source:
PNL spike ID number:
Anal. balance M&TE: 360-06-01-016
Sample filtered (yes/no): no

(1) Process factor = Final volume (ml) / [Vial & sample weight (g) - Vial weight (g)]

Other sample preparation worksheets may be substituted at the discretion of the Cognizant Scientist. Use one worksheet per client.

Analyst/Date: [signature] 9/1/95 Reviewer/Date: [signature] 9/1/95

Rev. 2.0 7-28-95 JMR

Battelle Pacific Northwest Laboratories
Analytical Chemistry Laboratory
Shielded Analytical Laboratory

Shielded Analytical Laboratory
Bench Sheet

Client: Kurt Silvers WP Number: K28863

TI#/ARF: TI95-TWC-02 / ASR 2388 Procedure: PNL-ALO-129

95-07941 Segment 1 95-07946 QTR Segment 4B Soluability Test
SAMPLE IDENTIFICATION

95-7941-1+2 clear, but had a white colored film of solids on bottom of vials.

95-7946 1+2 slightly colored, more so at the top and cloudy; no solids settled in the bottom.

1 ml Pipet DIW @ 23°C Made an 11.01X Dilution

.9926g $\bar{x}$ = .9955 g
.9897g
.9995g S = .0048
1.0011g
.9945g RSD = .48%
Vol = .9980 ml.

Fixed glass = 9.9903 ml. for DIW

M&TE:

✓ Mettler AE160 (360-06-01-016) Mettler AE200 (362-06-01-038) ___

___ Sartorius R200D (360-06-01-024) Corning pH Meter Model 240 S/N 6629 ___

Analyst: [signature] Date: 9/1/95 Reviewer: [signature] Date: 9/1/95

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WATER LEACH

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DATE TO QC: 09/19/95

DATA QUALITY REVIEW

I have reviewed the following data for completeness and for compliance with project requirements.

Analyte - Water Leach - Drainable Liquid

Data Package/Report - BY-108 Core 99

Project No. - 21372

ACL Numbers -	95-07932-C1	95-07932-C2	95-07932-C3	95-07932-C4
	95-07932-C5			
	95-07935-C1	95-07935-C2		

PNL ACL Quality Representative

9/20/95

Date

Battelle Pacific Northwest Laboratories
Analytical Chemistry Laboratory
Shielded Analytical Laboratory

Shielded Analytical Laboratory
WATER LEACH

Tank BY-108 Core(s) 99

Project Id: 21372 WP Number: K28864

TI Number: TI95-TWC-03 Procedure: PNL-ALO-103

	Sample Ident.	Sample Gross Wt (g)	Sample Tare Wt (g)	Sample Net Wt (g)	DIW Gross Wt (g)	DIW Tare Wt (g)	DIW Net Wt (g)	Spike Volume ml
	95-07932-C1 SEG 2 DL	21.8166	21.5311	0.2855	32.8628	13.7781	19.0847	
	95-07932-C2 SEG 2 DL DUP	21.6743	21.3791	0.2952	33.7486	13.4535	20.2951	
	95-07932-C3 METHODS BLK	N/A	N/A	N/A	41.3352	21.4985	19.8367	
MS	95-07932-C4 SEG 2 DL SPIKE	21.7945	21.5113	0.2832	33.6736	14.1280	19.5456	.1996
	95-07932-C5 BLANK SPIKE	N/A	~~21.5088~~ kp 9/7/95	N/A	34.2553	13.5922	20.6631	.1996
	95-07935-C1 SEG 3 DL	21.8441	21.5552	0.2889	33.6416	13.6214	20.0202	
	95-07935-C2 SEG 3 DL DUP	21.8552	21.5756	0.2796	33.9918	13.5510	20.4408	

Both segments had observable solids in the bottom of jar. I aliquoted only the liquid portion.

200λ Pipe
DIW @ 25°C
.1995 x̄ = .19
.1984 S = .00
.1996
.1991
.1994
Del = .1996

Spike Id: # 950831 ICP1

Ave. wt = 0.2882 g. using 4 weights.

M&TE:

✓ Mettler AE160 (360-06-01-016) ___ Mettler AE200 (362-06-01-038)

___ Sartorius R200D (360-06-01-024)

Analyst: [signature] Date: 9/7/95 Reviewer: [signature] Date: 9/8/95

Battelle Pacific Northwest Laboratories
Analytical Chemistry Laboratory
325 Shielded Analytical Laboratory

SAMPLE PREP SHEET
(325 SHIELDED ANALYTICAL LABORATORY)

TI/ARF NO.: TI95-TWC-03/ASR 2388 PROJECT NO.: 21732 WBS NO.: ______ SAMPLE TYPE: LIQUID

ISSUED BY: RT STEELE DATE: 8/31/95 PREP TYPE: Water Leach

ANALYST: [signature] DATE: 9/7/95 CONTROLLING PROCEDURE: PNL-ALO-110

REVIEW: [signature] DATE: 9/8/95 QA PLAN: MCS-033 IMPACT LEVEL: II

CLIENT: KURT SILVERS CORE ID: 99 TANK ID: BY-108

WORK PACKAGE NUMBER	ALO NUMBER	PROCEDURE	ANALYTE OR ANALYSIS	SAMPLE WT	WATER WT (g)	TOTAL VOL (mL)	SPIKE ID	SPIKE VOL (mL)	DILUTION FACTOR	DILUTION MATRIX	PIPET CALIB (mL)	MISC
K28867	95-07932-C1	PNL-ALO-212	IC	0.2855	19.0847						.1996	SAMP
	95-07932-C2		TOC RG 9/20/95	0.2952	20.2951							DUP
	95-07932-C3			—	19.8367							BLANK
	95-07932-C4			0.2832	19.5456		950831ICS01	.1996				MS
	95-07932-C5			—	20.6631							BLK SPK
	95-07935-C1			0.2889	20.0202							SAMP
	95-07935-C2			0.2796	20.4408							DUP

WHC-SD-WM-DP-145, REV. 1

Battelle Pacific Northwest Laboratories
Analytical Chemistry Laboratory
325 Shielded Analytical Laboratory

SAMPLE PREP SHEET
(325 SHIELDED ANALYTICAL LABORATORY)

TI/ARF NO.: TI95-TWC-03/ASR 2388 PROJECT NO.: 21732 WBS NO.: ______ SAMPLE TYPE: LIQUID

ISSUED BY: RT STEELE DATE: 8/31/95 PREP TYPE: Water Leach

ANALYST: [signature] DATE: 9/7/95 CONTROLLING PROCEDURE: PNL-ALO-110

REVIEW: [signature] DATE: 9/8/95 QA PLAN: MCS-033 IMPACT LEVEL: II

CLIENT: KURT SILVERS CORE ID: 99 TANK ID: BY-108

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WHC-SD-WM-DP-145, REV. 1

WORK PACKAGE NUMBER	ALO NUMBER	PROCEDURE	ANALYTE OR ANALYSIS	SAMPLE WT	WATER WT (g)	TOTAL VOL (mL)	SPIKE ID	SPIKE VOL (mL)	DILUTION FACTOR	DILUTION MATRIX	PIPET CALIB (mL)	MISC
K28866 ~~K28666~~	95-07932-C1	PNL-ALO-211	ICP	0.2855	19.0847							SAMP
RP Duke	95-07932-C2			0.2952	20.2951							DUP
	95-07932-C3		(RTS	9-8-95)	~~[illegible]~~	19.8367						BLANK
	95-07935-C1			0.2897	20.0202							SAMP
	95-07935-C2			0.2796	20.4408							DUP

DATE TO QC: 09/19/95

DATA QUALITY REVIEW

I have reviewed the following data for completeness and for compliance with project requirements.

Analyte - Water Leach

Data Package/Report - BY-108 Core 99

Project No. - 21372

ACL Numbers -	95-07941-C1	95-07941-C2	95-07941-C3	95-07941-C4
	95-07941-C5			
	95-07942-C1	95-07942-C2		
	95-07943-C1	95-07943-C2		
	95-07944-C1	95-07944-C2		
	95-07945-C1	95-07945-C2		
	95-07946-C1	95-07946-C2		
	95-07947-C1	95-07947-C2		
	95-07948-C1	95-07948-C2		

PNL ACL Quality Representative — 9/20/95 Date

Battelle Pacific Northwest Laboratories
Analytical Chemistry Laboratory
Shielded Analytical Laboratory

Shielded Analytical Laboratory
WATER LEACH

Tank BY-108 Core(s) 99

Project Id: 21372 WP Number: K28864

TI Number: TI95-TWC-03 Procedure: PNL-ALO-103

Sample Ident.	Sample Gross Wt (g)	Sample Tare Wt (g)	Sample Net Wt (g)	DIW Gross Wt (g)	DIW Tare Wt (g)	DIW Net Wt (g)	Spike ~~Volume~~ g.
95-07941-C1 SEG 1	91.6629	91.1490	0.5139	89.0226	38.8388	50.1838	
95-07941-C2 SEG 1 DUP	83.1335	82.6363	0.4972	90.2340	40.2525	49.9815	
95-07941-C3 METHODS BLK*	XXX	74.8676		125.3482	74.8676	50.4806	
95-07941-C4 MATRIX SPIKE	84.6592	84.1619	0.4973	94.5467	42.6344	51.9123	85.1769 84.6592 0.5177
95-07941-C5 SPIKE CONT.	85.8571	85.3388	0.5183	89.5364	38.7847	50.7517	
95-07942-C1 QTR SEG 2A	86.8215	86.3063	0.5152	92.2424	41.9017	50.3407	
95-07942-C2 QTR SEG 2A DUP	84.7279	84.2228	0.5051	92.8365	41.6529	51.1836	
95-07943-C1 QTRA SEG 2D	86.1853	85.6115	0.5738	96.3848	43.1204	53.2644	
95-07943-C2 QTR SEG 2D DUP	89.4940	88.8780	0.6160	92.6429	41.8188	50.8241	

* The me[thods] blan[k] fe[ll] ... H_2O ... but b... visual inspec... the inte... of the seal wa... not bro... ETB 9/6

Spike Id: Tank spike for SAL Prep. Spike Id 950831ICSP1

M&TE:

✓ Mettler AE160 (360-06-01-016) ___ Mettler AE200 (362-06-01-038)

___ Sartorius R200D (360-06-01-024)

Analyst: [signature] Date: 9/6/95 Reviewer: [signature] Date: 9/7/95

Battelle Pacific Northwest Laboratories
Analytical Chemistry Laboratory
Shielded Analytical Laboratory

Shielded Analytical Laboratory
WATER LEACH

Tank BY-108 Core(s) 99

Project Id: 21372 WP Number: K28864

TI Number: TI95-TWC-03 Procedure: PNL-ALO-103

Sample Ident.	Sample Gross Wt (g)	Sample Tare Wt (g)	Sample Net Wt (g)	DIW Gross Wt (g)	DIW Tare Wt (g)	DIW Net Wt (g)	Spike Volume
95-07944-C1 QTR SEG 3A	86.3989	85.8747	0.5242	94.7222	43.4411	51.2811	
95-07944-C2 QTR SEG 3A DUP	89.2134	88.7311	0.4823	93.8675	42.5354	51.3321	
95-07945-C1 QTR SEG 4A	87.1099	86.6210	0.4889	93.8845	42.3392	51.5453	
95-07945-C2 QTR SEG 4A DUP	90.2466	89.6430	0.6035	88.8333	38.8147	50.0186	
95-07946-C1 QTR SEG 4B	89.4803	89.0818	0.3985	90.3757	40.3788	49.9969	
95-07946-C2 QTR SEG 4B DUP	87.1453	86.5840	0.5613	92.4692	41.4173	51.0519	
95-07947-C1 QTR SEG 4C	81.1101	80.6485	0.4616	90.9456	40.4098	50.5358	
95-07947-C2 QTRA SEG 4C DUP	89.9728	89.4526	0.5202	90.2248	39.9628	50.2620	

Spike Id: ____________________

M&TE:

✓ Mettler AE160 (360-06-01-016) ___ Mettler AE200 (362-06-01-038)

___ Sartorius R200D (360-06-01-024)

Analyst: [signature] Date: 9/6/95 Reviewer: [signature] Date: 9/7/95

Battelle Pacific Northwest Laboratories
Analytical Chemistry Laboratory
Shielded Analytical Laboratory

Shielded Analytical Laboratory
WATER LEACH

Tank BY-108 Core(s) 99

Project Id: 21372 WP Number: K28864

TI Number: TI95-TWC-03 Procedure: PNL-ALO-103

Sample Ident.	Sample Gross Wt (g)	Sample Tare Wt (g)	Sample Net Wt (g)	DIW Gross Wt (g)	DIW Tare Wt (g)	DIW Net Wt (g)	Spike Volume
95-07948-C1 QTR SEG 4D	88.5036	88.0085	0.4951	93.9045	42.1162	51.7883	
95-07948-C2 QTR SEG 4D DUP	87.1724	86.5109	0.6615	91.2758	39.4423	51.8335	
0.5 ml pipet calibration for spike addition							
				DIW @ 24°C			
0.4910 g	$\bar{X}$ =	0.4923					
0.4898 g	S =	.0022					
0.4952 g	RSD =	0.44%					
0.4938 g	n =	5					
0.4917 g	DIW	.4936 ml					

Spike Id: ____________________

M&TE:

✓ Mettler AE160 (360-06-01-016) ___ Mettler AE200 (362-06-01-038)

___ Sartorius R200D (360-06-01-024)

Analyst: [signature] Date: 9/6/95 Reviewer: [signature] Date: 9/7/95

Battelle Pacific Northwest Laboratories
Analytical Chemistry Laboratory
325 Shielded Analytical Laboratory

SAMPLE PREP SHEET
(325 SHIELDED ANALYTICAL LABORATORY)

TI/ARF NO.: TI95-TWC-03/ASR 2388 PROJECT NO.: 21732 WBS NO.: ______ SAMPLE TYPE: SLUDGE

ISSUED BY: RT STEELE DATE: 8/31/95 PREP TYPE: Water Leach

ANALYST: Rick T. Steele DATE: 9/7/95 CONTROLLING PROCEDURE: PNL-ALO-110

REVIEW: [signature] DATE: 9/7/95 QA PLAN: MCS-033 IMPACT LEVEL: II

CLIENT: KURT SILVERS CORE ID: 99 TANK ID: BY-108

WORK PACKAGE NUMBER	ALO NUMBER	PROCEDURE	ANALYTE OR ANALYSIS	SAMPLE WT	WATER WT (g)	TOTAL VOL (mL)	SPIKE ID	SPIKE VOL (mL)	DILUTION FACTOR	DILUTION MATRIX	PIPET CALIB (mL)	MISC
K28866 ~~K28868~~	95-07941-C1	PNL-ALO-211	ICP		0.5139	50.1838						SAMP
	95-07941-C2				0.4972	49.9815						DUP
	95-07941-C3					50.4806						BLANK
	95-07942-C1				0.5152	50.3407						SAMP
	95-07942-C2				0.5051	51.1836						DUP
	95-07943-C1				0.5738	53.2644						SAMP
	95-07943-C2				0.6160	50.8241						DUP
	95-07944-C1				0.5242	51.2811						SAMP
	95-07944-C2				0.4823	51.3321						DUP
	95-07945-C1				0.4887	51.5453						SAMP
	95-07945-C2				0.6835	50.0186						DUP
	95-07946-C1				0.3985	49.9969						SAMP
	95-07946-C2				0.5613	51.6519						DUP
	95-07947-C1				0.4616	50.5358						SAMP

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WHC-SD-WM-DP-145, REV. 1

Battelle Pacific Northwest Laboratories
Analytical Chemistry Laboratory
325 Shielded Analytical Laboratory

SAMPLE PREP SHEET
(325 SHIELDED ANALYTICAL LABORATORY)

TI/ARF NO.: TI95-TWC-03/ASR 2388 PROJECT NO.: 21732 WBS NO.: ________ SAMPLE TYPE: SLUDGE

ISSUED BY: RT STEELE DATE: 8/31/95 PREP TYPE: Water Leach

ANALYST: Rick S. Steele DATE: 9/7/95 CONTROLLING PROCEDURE: PNL-ALO-110

REVIEW: [signature] DATE: 9/7/95 QA PLAN: MCS-033 IMPACT LEVEL: II

CLIENT: KURT SILVERS CORE ID: 99 TANK ID: BY-108

WORK PACKAGE NUMBER	ALO NUMBER	PROCEDURE	ANALYTE OR ANALYSIS	SAMPLE WT	WATER WT (g)	TOTAL VOL (mL)	SPIKE ID	SPIKE VOL (mL)	DILUTION FACTOR	DILUTION MATRIX	PIPET CALIB (mL)	MISC
K28866 ~~K28868~~ KP 9/11/95	95-07947-C2	PNL-ALO-211	ICP	0.5202	50.2620							DUP
	95-07948-C1			0.4951	51.7883							SAMP
	95-07948-C2			0.6615	51.8335							DUP

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WHC-SD-WM-DP-145, REV. 1

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Battelle Pacific Northwest Laboratories
Analytical Chemistry Laboratory
325 Shielded Analytical Laboratory

SAMPLE PREP SHEET
(325 SHIELDED ANALYTICAL LABORATORY)

TI/ARF NO.: TI95-TWC-03/ASR 2388 PROJECT NO.: 21732 WBS NO.: ______ SAMPLE TYPE: SLUDGE

ISSUED BY: RT STEELE DATE: 8/31/95 PREP TYPE: Water Leach

ANALYST: Rick T. Steele DATE: 9/7/95 CONTROLLING PROCEDURE: PNL-ALO-110

REVIEW: [signature] DATE: 9/7/95 QA PLAN: MCS-033 IMPACT LEVEL: II

CLIENT: KURT SILVERS CORE ID: 99 TANK ID: BY-108

WHC-SD-WM-DP-145, REV. 1

WORK PACKAGE NUMBER	ALO NUMBER	PROCEDURE	ANALYTE OR ANALYSIS	SAMPLE WT (g)	WATER WT (g)	TOTAL VOL (mL)	SPIKE ID	SPIKE ~~(mL)~~ wt (g)	DILUTION FACTOR	DILUTION MATRIX	PIPET CALIB (mL)	MISC
K28867	95-07941-C1	PNL-ALO-212	IC	0.5139	50.1838						0.4936	SAMP
	95-07941-C2			0.4972	49.9815							DUP
	95-07941-C3				50.4806							BLANK
	95-07941-C4			0.4973	51.9123		950831ICSP	0.5177				MS
	95-07941-C5		RTS 9-7-95	~~0.6183~~	50.7517			0.5183				SPK BLK
	95-07942-C1			0.5152	50.3407							SAMP
	95-07942-C2			0.5051	51.1836							DUP
	95-07943-C1			0.5738	53.2644							SAMP
	95-07943-C2			0.6160	50.8241							DUP
	95-07944-C1			0.5242	51.2811							SAMP
	95-07944-C2			0.4823	51.3321							DUP
	95-07945-C1			0.4889	51.5453							SAMP
	95-07945-C2			0.6035	50.0186							DUP
	95-07946-C1			0.3985	49.9969							SAMP
	95-07946-C2			0.5613	51.0519							DUP
	95-07947-C1			0.4616	50.5358							SAMP

Battelle Pacific Northwest Laboratories
Analytical Chemistry Laboratory
325 Shielded Analytical Laboratory

SAMPLE PREP SHEET
(325 SHIELDED ANALYTICAL LABORATORY)

TI/ARF NO.: TI95-TWC-03/ASR 2388 PROJECT NO.: 21732 WBS NO.: ______ SAMPLE TYPE: SLUDGE

ISSUED BY: RT STEELE DATE: 8/31/95 PREP TYPE: Water Leach

ANALYST: Rick T. Steele DATE: 9/7/95 CONTROLLING PROCEDURE: PNL-ALO-110

REVIEW: [signature] DATE: 9/2/95 QA PLAN: MCS-033 IMPACT LEVEL: II

CLIENT: KURT SILVERS CORE ID: 99 TANK ID: BY-108

WORK PACKAGE NUMBER	ALO NUMBER	PROCEDURE	ANALYTE OR ANALYSIS	SAMPLE WT (g)	WATER WT (g)	TOTAL VOL (mL)	SPIKE ~~ID~~ Wt (g) KH 9/7/95	SPIKE VOL (mL)	DILUTION FACTOR	DILUTION MATRIX	PIPET CALIB (mL)	MISC
K28867	95-07947-C2	PNL-ALO-212	IC	0.5207	50.2620							DUP
	95-07948-C1			0.4951	51.7883							SAMP
	95-07948-C2			0.6615	51.8335							DUP

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WHC-SD-WM-DP-145, REV. 1

ACID DIGESTION

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WHC-SD-WM-DP-145, REV. 1

DATE TO QC: 09/19/95

DATA QUALITY REVIEW

I have reviewed the following data for completeness and for compliance with project requirements.

Analyte - Acid Digest (Drainable Liquid)

Data Package/Report - BY-108 Core 99

Project No. - 21372

ACL Numbers - 95-07932-A1 95-07932-A2 95-07932-A3 95-07932-A4
95-07932-A5
95-07935-A1 95-07935-A2

PNL ACL Quality Representative

9/20/95
Date

Battelle Pacific Northwest Laboratories
Analytical Chemistry Laboratory
325 SAL-SRPL

PNL-ALO-128

Nitric and Hydrochloric Acid Extraction of High-level Radioactive Liquids and TCLP Leachates

Client: K. SILVERS
QA plan: MCS-033 [redacted]
Work Auth. Doc (WAD): ASR 2388 TI95-TWC-03
Impact level: II [redacted]
Work Package/Project: K28866
Balance M&TE: [redacted]
Tank/Core/Other ID: TANK BY-108 CORE 99 DRAINABLE LIQUIDS
Prep. lab (SAL or SRPL): SAL [redacted]
Additional Information: [redacted]

	ALO #	Client ID	Sample weight (g)	Spike volume (ml)	Final extract volume (ml)	Process Factor			
1	95-07932-A1	SEG 2 DL	2.7817		25	8.99			
2	95-07932-A2	SEG 2 DL DUP	2.7705			9.02			
3	95-07932-A3	METHODS BLANK	~~2.8757~~						
4	95-07932-A4	MATRIX SPIKE	2.8757	0.2526		8.69			
5	95-07932-A5	SPIKE CONTROL		0.2526					
6	95-07935-A1	SEG 3 DL	2.8016			8.92			
7	95-07935-A2	SEG 3 DL DUP	2.8259			8.85			
8									
9									
10									
11									
12									
13									
14									
15									
16									

Analyst comments (e.g. identification number for spikes, sample preparation problems encountered, unusual sample properties):

SAMPLES FILTERED: YES [ ] NO [ ]

SPike ID #950830H901 ICP AES .250 ml Pipet DIW @ 25°C
.2525 $\bar{x}$ = .2519
.2520 s = .0004
.2514 RSD = .17%
.2513
.2519 Del = .2526 ml

Analyst/Date: [signature] 9/8/95 Reviewer/Date: [signature] 9/8/95

rev.2.0 3-9-95 JMR

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WHC-SD-WM-DP-145, REV. 1

White flocculant noted upon acid addition

250 ul pipet check

5ml Pipet Check (GEA) DIW @ 25°C

~~4.9747~~ Void 9-8-95	4.9845	
4.9861	4.9547	$\bar{X}$ = 4.9812
4.9649	4.9917	S = 0.0175
4.9553	5.0001	RSD = 0.35%
4.9845	4.9750	n = 5
		DLVY = 4.9959 mL

Analyst: [signature] Date: 9/8/95

Reviewer: [signature] Date: 9/19/95

Battelle Pacific Northwest Laboratories
Analytical Chemistry Laboratory
325 Shielded Analytical Laboratory

SAMPLE PREP SHEET
(325 SHIELDED ANALYTICAL LABORATORY)

TI/ARF NO.: TI95-TWC-03/ASR 2388 PROJECT NO.: 21732 WBS NO.: ______ SAMPLE TYPE: DRAINABLE LIQUID

ISSUED BY: RT STEELE DATE: 8/31/95 PREP TYPE: ACID DIGESTION

ANALYST: [signature] DATE: 9/8/95 CONTROLLING PROCEDURE: PNL-ALO-110

REVIEW: [signature] DATE: 9/11/95 QA PLAN: MCS-033 IMPACT LEVEL: II

CLIENT: KURT SILVERS CORE ID: 99 TANK ID: BY-108

WORK PACKAGE NUMBER	ALO NUMBER	PROCEDURE	ANALYTE OR ANALYSIS	SAMPLE WT	WATER WT (g)	TOTAL VOL (mL)	SPIKE ID	SPIKE VOL (mL)	DILUTION FACTOR	DILUTION MATRIX	PIPET CALIB (mL)	MISC
K28866	95-07932-A1	PNL-ALO-211	ICP									SAMP
	95-07932-A2											DUP
	95-07932-A3											BLANK
	95-07932-A4											MS
	95-07932-A5											SPK CONT
	95-07935-A1											SAMP
	95-07935-A2											DUP

PAGE 1 OF 1

WHC-SD-WM-DP-145, REV. 1

Battelle Pacific Northwest Laboratories
Analytical Chemistry Laboratory
325 Shielded Analytical Laboratory

SAMPLE PREP SHEET
(325 SHIELDED ANALYTICAL LABORATORY)

TI/ARF NO.: TI95-TWC-03/ASR 2388 PROJECT NO.: 21732 WBS NO.: ______ SAMPLE TYPE: DRAINABLE LIQUID

ISSUED BY: RT STEELE DATE: 8/31/95 PREP TYPE: ACID DIGESTION

ANALYST: [signature] DATE: 9/8/95 CONTROLLING PROCEDURE: PNL-ALO-110

REVIEW: [signature] DATE: 9/11/95 QA PLAN: MCS-033 IMPACT LEVEL: II

CLIENT: KURT SILVERS CORE ID: 99 TANK ID: BY-108

WORK PACKAGE NUMBER	ALO NUMBER	PROCEDURE	ANALYTE OR ANALYSIS	SAMPLE WT	WATER WT (g)	TOTAL VOL (mL)	SPIKE ID	SPIKE VOL (mL)	DILUTION FACTOR	DILUTION MATRIX	PIPET CALIB (mL)	MISC
SEE BELOW	95-07932-A1	SEE BELOW	SEE BELOW									samp
	95-07932-A2											dup
	95-07932-A3											blank
	95-07935-A1											samp
	95-07935-A2											dup
K28870		PNL-ALO-421	T. ALPHA									
K28872		PNL-ALO-450	GEA									
K28873		PNL-ALO-431	T. BETA									

You received 9.9919 mL of each of samples above.

KOH FUSION

DATE TO QC: 09/19/95 WHC-SD-WM-DP-145, REV. 1

DATA QUALITY REVIEW

I have reviewed the following data for completeness and for compliance with project requirements.

Analyte - KOH Fusion

Data Package/Report - BY-108 Core 99

Project No. - 21372

ACL Numbers -	95-07942-H1	95-07942-H2	95-07942-H3
	95-07943-H1	95-07943-H2	
	95-07944-H1	95-07944-H2	
	95-07945-H1	95-07945-H2	95-07945-H3
	95-07947-H1	95-07947-H2	
	95-07948-H1	95-07948-H2	

[signature]
PNL ACL Quality Representative

9/20/95
Date

Battelle Pacific Northwest Laboratories
Analytical Chemistry Laboratory
Shielded Analytical Laboratory

Shielded Analytical Laboratory
FUSIONS

Tank BY-108 Core(s) 99

Project Id: TWC/21372 WP Number: K28862

Test Instruction: TI95-TWC-03 Procedure: PNL-ALO-115

	Sample Ident.	Wet Sample Gross Wt (g)	Wet Sample Tare Wt (g)	Wet Sample Net Wt (g)	Dry Sample Gross Wt (g)	Dry Sample Tare Wt (g)	Dry Sample Net Wt (g)
1	95-07942-H1 QTR SEG 2A	54.7145	54.5993	0.1152			
2	95-07942-H2 QTR SET 2A DUP	48.7459	48.6350	0.1109			
3	95-07942-H3 METHODS BLANK	XXXX	XXXX				
4	95-07943-H1 QTR SEG 2D	54.3861	54.0499	0.3362			
5	95-07943-H2 QTR SEG 2D DUP	55.4510	55.1247	0.3263			
6	95-07944-H1 QTR SEG 3A	55.7204	55.5992	0.1212			
7	95-07944-H2 QTR SEG 3A DUP	54.4753	54.3333	0.1420			

M&TE:

X Mettler AE160 (360-06-01-016) ___ Mettler AE200 (362-06-01-038)

___ Sartorius R200D (360-06-01-024)

Analyst: [signature] Date: 9/11/95 Reviewer: [signature] Date: 9/11/95

Battelle Pacific Northwest Laboratories
Analytical Chemistry Laboratory
Shielded Analytical Laboratory

Shielded Analytical Laboratory
FUSIONS

Tank BY-108 Core(s) 99

Project Id: TWC/21372 WP Number: K28862

Test Instruction: TI95-TWC-03 Procedure: PNL-ALO-115

	Sample Ident.	Wet Sample Gross Wt (g)	Wet Sample Tare Wt (g)	Wet Sample Net Wt (g)	Dry Sample Gross Wt (g)	Dry Sample Tare Wt (g)	Dry Sample Net Wt (g)
✓8	95-07945-H1 QTR SEG 4A*	53.3293	53.2216	0.1077			
9	95-07945-H2 QTR SET 4A DUP	56.4366 ~~56.4613~~ 9/11/95 OTS	56.3194 ~~56.2912~~ 9/11/15 OTS	0.1172 ~~0.1701~~* 9/11/95 OTS			
10	95-07945-H3 METHODS BLANK						
✓ 11	95-07947-H1 QTR SEG 4C	55.7426	55.6337	0.1089			
✓ 12	95-07947-H2 QTR SEG 4C DUP	54.1167 ~~54.0877~~	OTS 9/11/15 53.9722	0.1445			
13	95-07948-H1 QTR SEG 4D	52.8227	52.7239	0.0988			
14	95-07948-H2 QTR SEG 4D DUP	54.9214	54.8338	0.0876			
#15	*SRM 2709	54.1551	54.0560	0.0991			

* started pink yellow in color, put on the plate and pink in color + put on the plate the solution boil over (will need to be repeated) OTS 9/7

M&TE:

___ Mettler AE160 (360-06-01-016) ___ Mettler AE200 (362-06-01-038)

___ Sartorius R200D (360-06-01-024)

Analyst: [signature] Date: 9/6/95 Reviewer: [signature] Date: 9/11/95

* SRM 2709 Barcode # 80804

Battelle Pacific Northwest Laboratories
Analytical Chemistry Laboratory
325 Shielded Analytical Laboratory

SAMPLE PREP SHEET
(325 SHIELDED ANALYTICAL LABORATORY)

TI/ARF NO.: TI95-TWC-03/ASR 2388 PROJECT NO.: 21732 WBS NO.: ______ SAMPLE TYPE: SLUDGE

ISSUED BY: RT STEELE DATE: 8/31/95 PREP TYPE: NI/KOH FUSION

ANALYST: [signature] DATE: 9/11/95 CONTROLLING PROCEDURE: PNL-ALO-110

REVIEW: [signature] DATE: 9/11/95 QA PLAN: MCS-033 IMPACT LEVEL: II

CLIENT: KURT SILVERS CORE ID: 99 TANK ID: BY-108

WORK PACKAGE NUMBER	ALO NUMBER	PROCEDURE	ANALYTE OR ANALYSIS	SAMPLE WT	WATER WT (g)	TOTAL VOL (mL)	SPIKE ID	SPIKE VOL (mL)	DILUTION FACTOR	DILUTION MATRIX	PIPET CALIB (mL)	MISC
SEE BELOW	95-07945-H1	SEE BELOW	SEE BELOW	0.1077		100						SAMP
	95-07945-H2			0.1172								DUP
	95-07945-H3											BLANK
	95-07946-H1T			0.1002								SAMP
	95-07946-H2T			0.0992								DUP
	95-07946-HCB											BLANK
	95-07947-H1			0.1089								SAMP
	95-07947-H2			0.1445								DUP
	95-07948-H1			0.0988								SAMP
	95-07948-H2			0.0876								DUP
K28870		PNL-ALO-421	T. ALPHA									
K28871		PNL-ALO-422/423	AEA									
K28872		PNL-ALO-450	GEA									
K28873		PNL-ALO-431	T. BETA									
K28874		PNL-ALO-433/431	Sr-90									
[illegible]8875		PNL-ALO-445	U-LASER									

You received 9.9839 ml of each sample

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WHC-SD-WM-DP-145, REV. 1

9/11/95 Battelle Pacific Northwest Laboratories
Analytical Chemistry Laboratory
Shielded Analytical Laboratory

5.0ml pipet calibration check (GEA)

~~4.8603 g~~ OPB 9/11/95
4.9688 g
4.9692 g
4.9822 g
4.9863 g
4.9815 g

$\bar{X}$ = 4.9772 g
S = 0.0086
RSD = 0.17%
n = 5
dlvy = 4.9919 ml

cell 2 temperature: 25 °C

Analyst: [signature] Date: 9/19/95

Reviewer [signature] Date: 9/19/95

Battelle Pacific Northwest Laboratories
Analytical Chemistry Laboratory
325 Shielded Analytical Laboratory

SAMPLE PREP SHEET
(325 SHIELDED ANALYTICAL LABORATORY)

TI/ARF NO.: TI95-TWC-03/ASR 2388 PROJECT NO.: 21732 WBS NO.: ____ SAMPLE TYPE: SLUDGE

ISSUED BY: RT STEELE DATE: 8/31/95 PREP TYPE: NI/KOH FUSION

ANALYST: [signature] DATE: 9/11/95 CONTROLLING PROCEDURE: PNL-ALO-110

REVIEW: [signature] DATE: 9/11/95 QA PLAN: MCS-033 IMPACT LEVEL: II

CLIENT: KURT SILVERS CORE ID: 99 TANK ID: BY-108

WORK PACKAGE NUMBER	ALO NUMBER	PROCEDURE	ANALYTE OR ANALYSIS	SAMPLE WT	WATER WT (g)	TOTAL VOL (mL)	SPIKE ID	SPIKE VOL (mL)	DILUTION FACTOR	DILUTION MATRIX	PIPET CALIB (mL)	MISC
SEE BELOW	95-07941-H1T	SEE BELOW	SEE BELOW	0.1005		100						SAMP
	95-07941-H2T			0.1026								DUP
	95-07941-HCB											BLANK
	95-07942-H1			0.1152								SAMP
	95-07942-H2			0.1109								DUP
	95-07942-H3											BLANK
	95-07943-H1			0.3362								SAMP
	95-07943-H2			0.3263								DUP
	95-07944-H1			0.1212								SAMP
	95-07944-H2			0.1420								DUP
K28870		PNL-ALO-421	T. ALPHA									
K28871		PNL-ALO-422/423	AEA			You received ~~9.98~~ 39 mL of						
K28872		PNL-ALO-450	GEA			each sample.						
K28873		PNL-ALO-431	T. BETA									
K28874		PNL-ALO-433/431	Sr-90									
K28875		PNL-ALO-445	U-LASER									

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WHC-SD-WM-DP-145, REV. 1

Battelle Pacific Northwest Laboratories
Analytical Chemistry Laboratory
325 Shielded Analytical Laboratory

SAMPLE PREP SHEET
(325 SHIELDED ANALYTICAL LABORATORY)

TI/ARF NO.: TI95-TWC-03/ASR 2388 PROJECT NO.: 21732 WBS NO.: ______ SAMPLE TYPE: SLUDGE

ISSUED BY: RT STEELE DATE: 8/31/95 PREP TYPE: Ni/KOH FUSION

ANALYST: [signature] DATE: 9/11/95 CONTROLLING PROCEDURE: PNL-ALO-110

REVIEW: [signature] DATE: 9/11/95 QA PLAN: MCS-033 IMPACT LEVEL: II

CLIENT: KURT SILVERS CORE ID: 99 TANK ID: BY-108

WORK PACKAGE NUMBER	ALO NUMBER	PROCEDURE	ANALYTE OR ANALYSIS	SAMPLE WT	WATER WT (g)	TOTAL VOL (mL)	SPIKE ID	SPIKE VOL (mL)	DILUTION FACTOR	DILUTION MATRIX	PIPET CALIB (mL)	MISC
K28866	95-07941-H1T	PNL-ALO-211	ICP	0.1005		100						SAMP
	95-07941-H2T			0.1026								DUP
	95-07941-HCB											BLANK
	95-07942-H1			0.1152								SAMP
	95-07942-H2			0.1109								DUP
	95-07942-H3											BLANK
	95-07943-H1			0.3362								SAMP
	95-07943-H2			0.3263								DUP
	95-07944-H1			0.1212								SAMP
	95-07944-H2			0.1420								DUP
	~~95-07941-H4~~ SRM-2109-H	RTS 9-11-95		0.0991		↓						

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WHC-SD-WM-DP-145, REV. 1

Battelle Pacific Northwest Laboratories
Analytical Chemistry Laboratory
325 Shielded Analytical Laboratory

SAMPLE PREP SHEET
(325 SHIELDED ANALYTICAL LABORATORY)

TI/ARF NO.: T195-TWC-03/ASR 2388 PROJECT NO.: 21732 WBS NO.: SAMPLE TYPE: SLUDGE

ISSUED BY: RT STEELE DATE: 8/31/95 PREP TYPE: NI/KOH FUSION

ANALYST: [signature] DATE: 9/4/95 CONTROLLING PROCEDURE: PNL-ALO-110

REVIEW: [signature] DATE: 9/11/95 QA PLAN: MCS-033 IMPACT LEVEL: II

CLIENT: KURT SILVERS CORE ID: 99 TANK ID: BY-108

WORK PACKAGE NUMBER	ALO NUMBER	PROCEDURE	ANALYTE OR ANALYSIS	SAMPLE WT	WATER WT (g)	TOTAL VOL (mL)	SPIKE ID	SPIKE VOL (mL)	DILUTION FACTOR	DILUTION MATRIX	PIPET CALIB (mL)	MISC
K28866	95-07945-H1	PNL-ALO-211	ICP	0.1077		100						SAMP
	95-07945-H2			0.1172								DUP
	95-07945-H3											BLANK
	95-07946-H1T			0.1002								SAMP
	95-07946-H2T			0.0992								DUP
	95-07946-HCB											BLANK
	95-07947-H1			0.1089								SAMP
	95-07947-H2			0.1445								DUP
	95-07948-H1			0.0988								SAMP
	95-07948-H2			0.0876								DUP
	~~95-04446-H1~~ RTS 9-11-95 SRM-2709-H			0.0991								

3-209

WHC-SD-WM-DP-145, REV. 1

Na_2O_2 FUSION

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DATE TO QC: 09/19/95

DATA QUALITY REVIEW

I have reviewed the following data for completeness and for compliance with project requirements.

Analyte - Zr Fusions

Data Package/Report - BY-108 Core 99

Project No. - 21372

ACL Numbers - 95-07941-N1 95-07941-N2 95-07941-N3
95-07942-N1
95-07943-N1
95-07944-N1
95-07945-N1
95-07946-N1
95-07947-N1
95-07948-N1

PNL ACL Quality Representative — 9/20/95 Date

Battelle Pacific Northwest Laboratories
Analytical Chemistry Laboratory
Shielded Analytical Laboratory

Shielded Analytical Laboratory
FUSIONS

Tank BY-108 Core(s) 99

Project Id: 21372 WP Number: K28863

Test Instruction: TI95-TWC-03 Procedure: PNL-ALO-114

	*36.3677 Sample Ident. 9-7-95	Wet Sample Gross Wt (g)	Wet Sample Tare Wt (g)	Wet Sample Net Wt (g)	Dry Sample Gross Wt (g)	Dry Sample Tare Wt (g)	Dry Sample Net Wt (g)	
1	95-07941-N1 SEG 1 YK 9-7-95	~~36.3866~~ * ~~36.3677~~ ~~36.3974~~	36.2354	0.1323 ~~0.1620~~ YK 9-7-95				hard small chunks run after NaOH addition, & dissolved
3	95-07941-N2 SEG 1 DUP	36.6589	36.5643	0.0946				ditto
4	95-07941-N3 METH BLANK		36.6181					okay
5	95-07942-N1 QTR SEG 2~~Y~~A YM	36.4918	36.3868	0.1050				okay
9	95-07943-N1 QTR SEG 2D	37.2343	37.0087	0.2256				okay
13	95-07944-N1 QTR SEG 3A	36.7272 ~~36.7112~~ GPB 9/7/95	36.5944	0.1328 ~~0.1168~~ GPB 9/7/95				Chunks of NaOH

M&TE:

X Mettler AE160 (360-06-01-016) ___ Mettler AE200 (362-06-01-038)

___ Sartorius R200D (360-06-01-024)

Analyst: [signature] Date: 9/11/95 Reviewer: [signature] Date: 9/11/95

Battelle Pacific Northwest Laboratories
Analytical Chemistry Laboratory
Shielded Analytical Laboratory

Shielded Analytical Laboratory FUSIONS

Tank BY-108 Core(s) 99

Project Id: 21372 WP Number: K28863

Test Instruction: T195-TWC-03 Procedure: PNL-ALO-114

	Sample Ident.	Wet Sample Gross Wt (g)	Wet Sample Tare Wt (g)	Wet Sample Net Wt (g)	Dry Sample Gross Wt (g)	Dry Sample Tare Wt (g)	Dry Sample Net Wt (g)	
14	95-07945-N1 QTR SEG 4A	37.4115	37.3077	0.1038				Chunks of NaOH
15	95-07946-N1 QTR SEG 4B	37.5889	37.4812	0.1077				Chunks of NaOH
17	95-07947-N1 QTR SEG 4C	37.2300	37.1254	0.1046				Chunks of NaOH
18	95-07948-N1 QTR SEG 4D	36.9533	36.8432	0.1101				Chunks of NaOH
19	SRM 2709	36.4637	36.3009	0.1628				Okay

M&TE:

X Mettler AE160 (360-06-01-016) ___ Mettler AE200 (362-06-01-038)

___ Sartorius R200D (360-06-01-024)

Analyst: [signature] Date: 9/11/95 Reviewer: [signature] Date: 9/11/95

Battelle Pacific Northwest Laboratories
Analytical Chemistry Laboratory
325 Shielded Analytical Laboratory

SAMPLE PREP SHEET
(325 SHIELDED ANALYTICAL LABORATORY)

TI/ARF NO.: TI95-TWC-03/ASR 2388 PROJECT NO.: 21732 WBS NO.: ________ SAMPLE TYPE: SLUDGE

ISSUED BY: RT STEELE DATE: 8/31/95 PREP TYPE: Zr/Na2O2 FUSION

ANALYST: [signature] DATE: 9/11/95 CONTROLLING PROCEDURE: PNL-ALO-110

REVIEW: Rock T. Steele DATE: 9/11/95 QA PLAN: MCS-033 IMPACT LEVEL: II

CLIENT: KURT SILVERS CORE ID: 99 TANK ID: BY-108

WORK PACKAGE NUMBER	ALO NUMBER	PROCEDURE	ANALYTE OR ANALYSIS	SAMPLE WT	WATER WT (g)	TOTAL VOL (mL)	SPIKE ID	SPIKE VOL (mL)	DILUTION FACTOR	DILUTION MATRIX	PIPET CALIB (mL)	MISC
K28866	95-07941-N1	PNL-ALO-211	ICP	0.1323		100						SAMP
	95-07941-N2			0.0946								DUP
	95-07941-N3											BLANK
	~~95-07942-N1~~	RS 9-11-95		~~0.1050~~								~~SAMP~~
	95-07943-N1			0.2256								SAMP
	95-07944-N1			0.1328								SAMP
	95-07945-N1			0.1038								SAMP
	95-07946-N1			0.1077								SAMP
	95-07947-N1			0.1046								SAMP
	95-07948-N1			0.1101								
	SRM 2709			0.1628								

PAGE 1 OF _

SINGLE SHELL TANK

WASTE CHARACTERIZATION PROJECT

APPENDIX B

PHYSICAL PROPERTIES

TANK BY-108 CORE 99

September 1995

Pacific Northwest Laboratory

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B1 - INTRODUCTION

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Introduction to Physical Properties Primary Data Package

This section of the BY-108 Core 99 Primary Data Package contains primary or "raw" data collected during preparation and analysis of physical properties on Core 99 samples.

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B2 - DSC/TGA ANALYSIS

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DATE TO QC: 09/29/95

DATA QUALITY REVIEW

I have reviewed the following data for completeness and for compliance with project requirements.

Analyte - DSC/TGA

Data Package/Report - BY-108 Core 99

Project No. - 21372

ACL Numbers - 7313 7313-2 95-07932 95-07932-2
7315 7315-2 7314 7314-2 95-07935
95-07935-2 7316 7316-2 7322 7322-2
7319 7319-2 7318 7318-2 7317 7313
7317-2

JD Matheson

PNL ACL Quality Representative

9-29-95

Date

Thermal Analysis

Scanning Thermogravimetric Analysis (TGA) and Differential Scanning Calorimetry (DSC) were performed on the unhomogenized solids and drainable liquids from Core 99 Segments 1 through 4. These two thermal analysis techniques are useful in determining the thermal stability and reactivity of the material. DSC measures heat released or absorbed while the temperature of the sample is increased at a constant rate. Data generated by the DSC analysis is often used to measure thermal decomposition temperatures, heats of reaction, reaction temperatures, melting points and solid-solid transition temperatures. TGA measures the mass of a sample while the temperature of the sample is increased at a constant rate. The TGA data is used to measure thermal decomposition temperatures, water content and reaction temperatures. Both methods can be modified to measure isothermal changes in the material and provide complimentary information.

The calibration of the DSC and TGA instruments were checked before running these samples. An indium standard was run on the DSC to check the temperature and enthalpy calibrations. The balance calibration of the TGA was checked with a 100 mg standard weight, and the temperature calibration was checked with a lead standard. The temperature and enthalpy calibration checks were all within 1°C and 0.2 J/g of their reported values, and the balance calibration was within 0.01 mg.

The results from the DSC and TGA analyses are reported in Tables 1-6 and 1-7, respectively. The temperature range of the scans was from ambient to 550°C, with a scan rate of 10°C per minute. These analyses were all performed in platinum pans under nitrogen cover gas.

The major transition in all of these samples was an endotherm due to water loss from the sample. This was also the major mass loss in all of the samples. The onset temperature of this transition could not be measured because the transition began at the initial temperature of the run (ambient temperature). The water loss endotherm ends at approximately 200°C. This endotherm is a complicated system of several unresolvable transitions with each sample containing slightly different proportions of these transitions.

An exotherm was identified in all quarter segment samples from Segment 4 and the drainable liquids. This exotherm has an onset temperature of between 197 and 225°C. The enthalpy of this transition varied with each quarter segment. The temperature range for this transition was from 195 to 400°C. It appears that this exotherm is commingled with some endothermic behavior which cannot be resolved; therefore, it is difficult to determine onset temperatures for each sample. The enthalpy of this transition is also dependent upon resolution of this exotherm for the commingled endotherms and the initial water loss endotherm. In most cases the most conservative approach (the largest exothermic energy) has been taken.

In Segments 1 through 3 a second endotherm is observed. Several transitions are observed in this endotherm and the onset temperature varies depending upon the size of the first transition in comparison with the second

Table 1-6: Tank BY-108, Core 99 Differential Scanning Calorimetric (DSC) Results

Sample Number	Segment ID	Enthalpy (J/g)	Range (°C)	Onset (°C)
7313	1	725.6 311.2	8-153 193-333	219
7313-2	1	521.2 305.2	16-158 181-343	221
95-07932	2-DL	737.5 -11.6 -14.9	45-226 226-265 265-340	278
95-07932-2	2-DL	1243.6 -35.8 -10.5	20-216 216-270 272-330	279
7315	2A	201.3 108.6	48-227 227-324	250
7315-2	2A	177.3 114.1	43-231 231-322	251
7314	2D	263.0 127.3 -2.8	22-204 215-332 375-430	261 385
7314-2	2D	194.7 161.7 -2.8	24-185 187-345 380-430	211
95-07935	3-DL	1178.2 26.1	31-236 236-303	237
95-07935-2	3-DL	1279.2 26.9	32-239 239-300	238
7316	3A	303.4 81.1 -1.2	28-230 236-336 400-427	249
7316-2	3A	343.5 101.1 -0.7	12-215 233-327 430-450	259
7322	4A	385.5 33.6	22-183 243-310	259
7322-2	4A	556.7	33-255	
7319	4B	651.6 -191.1	22-193 200-411	207

Table 1-6: Tank BY-108, Core 99 Differential Scanning Calorimetric (DSC) Results (Cont.)

Sample Number	Segment ID	Enthalpy (J/g)	Range (°C)	Onset (°C)
7319-2	4B	476.0 -133.2	19-224 224-385	
7318	4C	644.5 -67.0	26-192 192-375	197
7318-2	4C	876.6 -77.8	15-196 200-361	237
7317	4D	693.4 -73.7	23-195 205-369	206
7317-2	4D	613.9 -60.2	30-193 203-350	204

transition. It appears that the onset temperature for the first transition in this endotherm is 220°C, and the onset temperature for the second transition is between 250 and 260°C.

The TGA analysis also indicated two different waste types in this core sample. All of the segments had a large mass loss associated with the water loss endotherm. This mass loss was observed between ambient temperature and 180°C. A small mass loss was observed over the remainder of the temperature range of the TGA analysis for all of the Segment 4 samples, but no significant transitions were observed at higher temperatures. In the Segment 1 through 3 solid samples, a second significant mass loss was observed. This mass loss has an onset temperature of approximately 245°C. This transition correlates with the second endothermic transition observed in the DSC analyses. These samples also continued to have a small mass loss throughout the remainder of the temperature range.

Table 1-7: Tank BY-108, Core 99 Thermogravimetric Analysis (TGA) Results

Sample	Segment	Range (°C)	Onset (°C)	Mass Loss (%)
7313	1	23-157 157-309 309-548	247	32.7 9.7 2.6
7313-2	1	28-145 145-318 318-548	249	26.1 13.2 3.5
7316	3A	22-212 212-344 344-548	310	14.1 0.9 0.2
7316	3A	40-170 170-548		35.0 1.9
7314	2D	27-198 198-328 328-545	245	17.9 2.2 0.9
7314-2	2D	23-203 203-328 328-442 442-545	242 393	15.5 3.3 0.8 0.4
7315	2A	23-136 136-256 256-545		7.8 3.6 0.8
7315-2	2A	23-130 130-215 215-545		6.6 5.2 0.1
7317	4D	23-167 167-550		40.6 5.1
7317-2	4D	22-178 178-549		44.0 3.8
7318	4C	24-173 173-549		35.6 5.3
7318-2	4C	24-172 172-549		36.2 4.5
7319	4B	22-187 187-403 403-548	309	35.3 7.9 1.3

Table 1-7: Tank BY-108, Core 99 Thermogravimetric Analysis (TGA) Results (Cont.)

Sample	Segment	Range (°C)	Onset	Mass Loss (%)
7319-2	4B	23-172 172-412 412-549	300	35.9 8.1 0.8
7322	4A	22-160 160-550		25.8 3.1
7322-2	4A	23-171 171-548		25.1 3.4
95-07935	3-DL	23-227 227-548		52.1 0.8
95-07935	3-DL	24-235 235-548		52.6 0.7
95-07932	2-DL	28-218 218-548		52.6 0.8
95-07932	2-DL	23-236 236-547		46.7 0.6

DSC
<Name>
14sept95a
<Date>
95/09/14 06:20

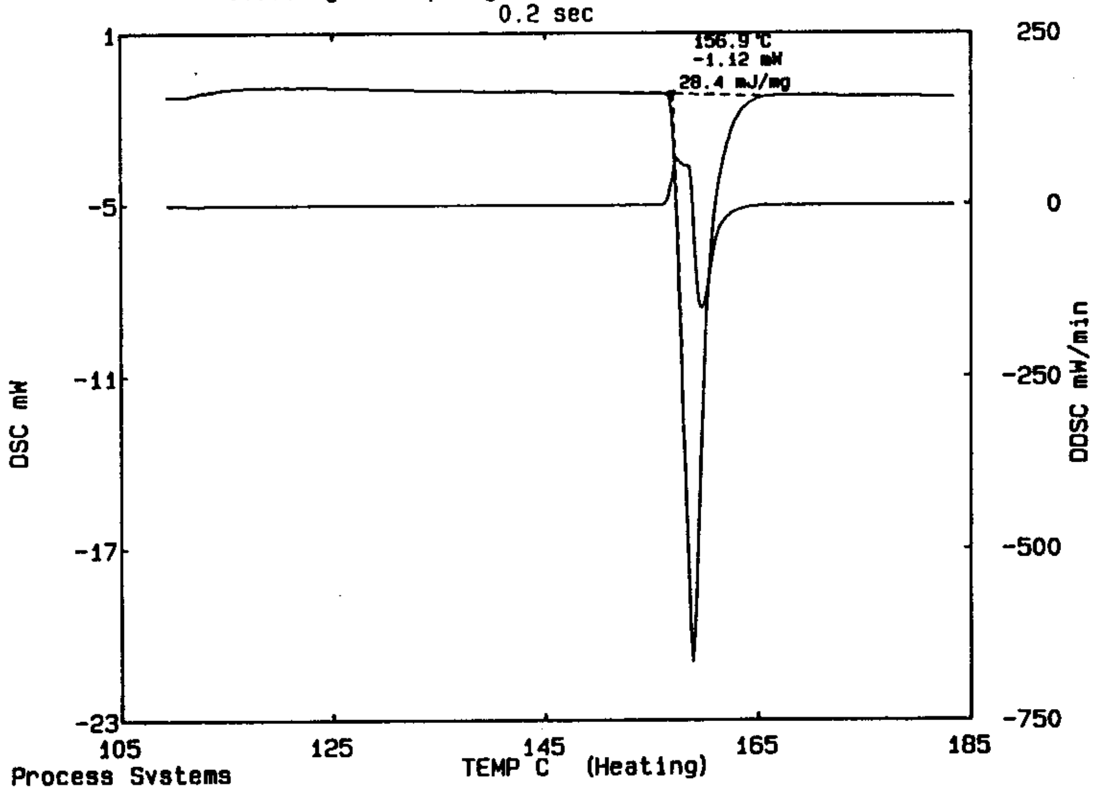
<Sample>
14sept95a
11.700 mg
(11.700 mg)
<Reference>
mt pan and lid
0.000 mg

<Comment>
indium std

<Sampling>
0.2 sec

<Temp.program[C]	[C/min]	[min]>
1 20.0- 120.0	20.00	0.00
2* 120.0- 190.0	10.00	0.00

<Gas>
nitrogen 300.0 ml/min
---------------- 0.0 ml/min



Chemical Process Systems

156.6
28.59

3-227

WHC-SD-WM-DP-145, REV. 1

TG/DTA

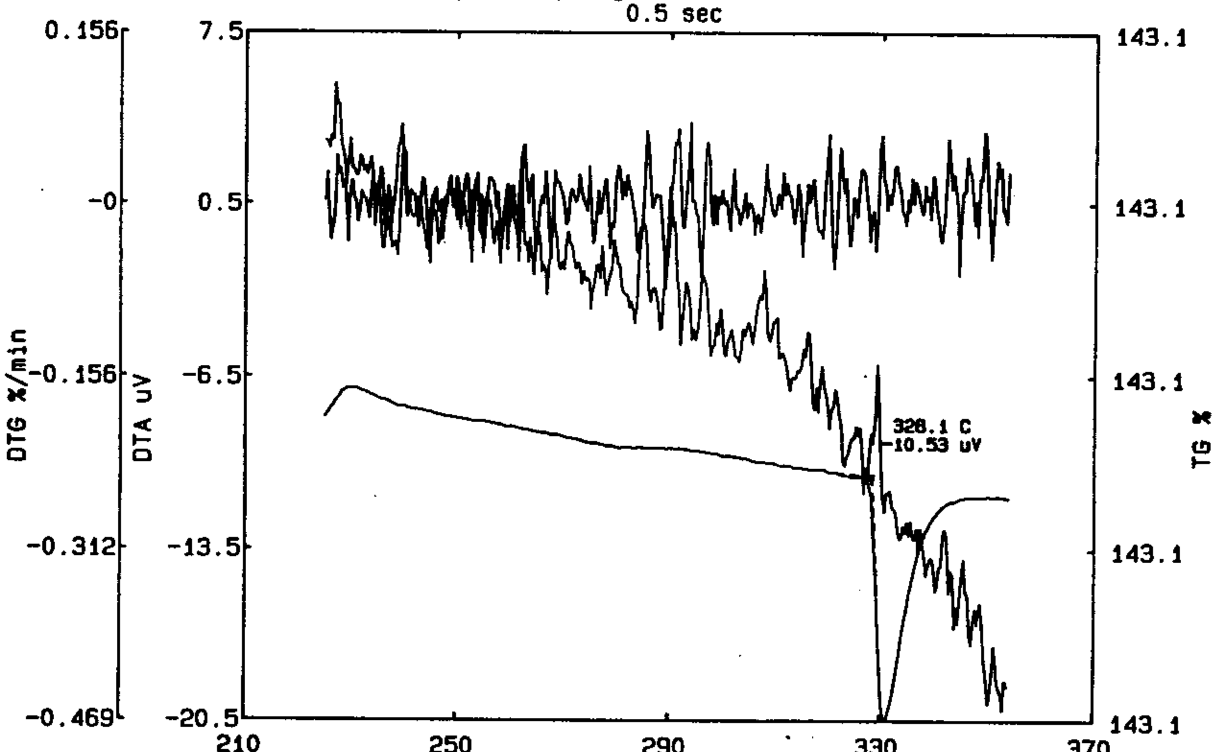
<Name>
14sept95d
<Date>
95/09/14 06:48

<Sample>
14sept95d
19.200 mg
(19.200 mg)
<Reference>
mt pan
0.000 mg

<Comment>
lead std
<Sampling>
0.5 sec

<Temp.program [C]		[C/min]	[min]>
1	20.0- 250.0	25.00	1.00
2*	250.0- 370.0	10.00	0.00

<Gas>
nitrogen 300.0 ml/min
0.0 ml/min



Chemical Process Systems

327.5
DKa

WHC-SD-WM-DP-145, REV. 1

3-228

DSC

<Name>
20sept95c

<Date>
95/09/20 08:38

<Sample>
20sept95c
19.935 mg
(19.935 mg)

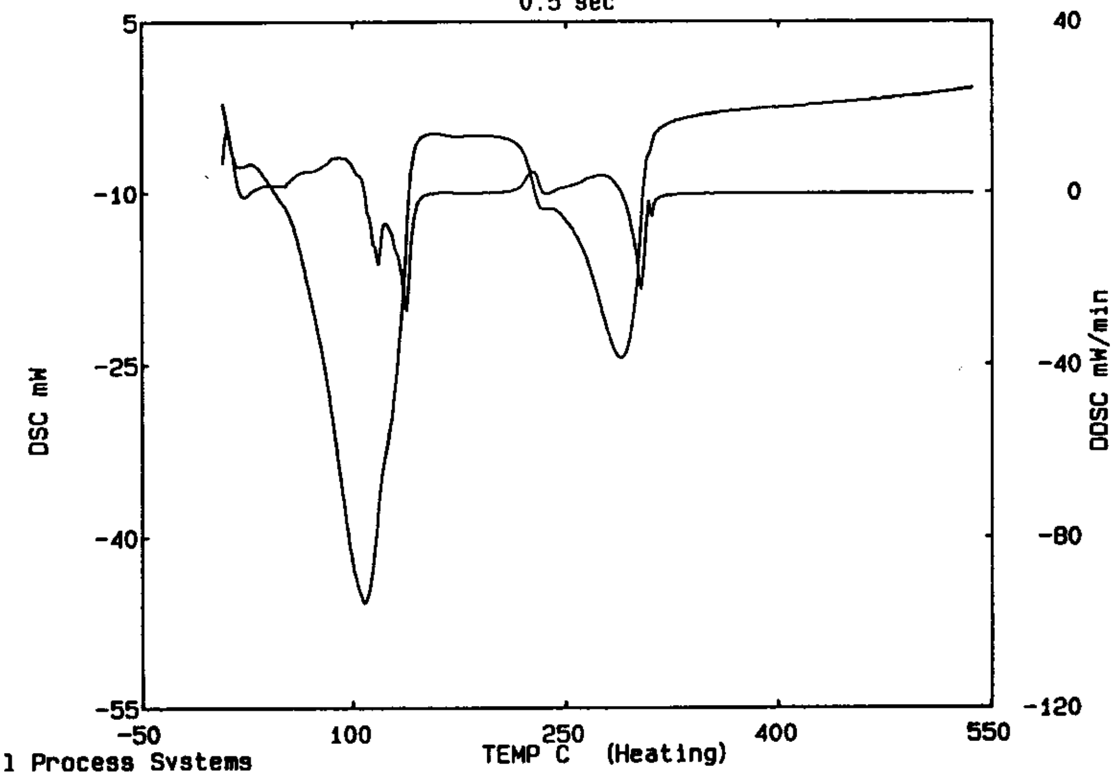
<Reference>
pt pan
0.000 mg

<Comment>
7313

<Sampling>
0.5 sec

<Temp.program[C] [C/min] [min]>
1* 20.0- 550.0 10.00 0.00

<Gas>
nitrogen 300.0 ml/min
0.0 ml/min



Chemical Process Systems

3-229

WHC-SD-WM-DP-145, REV. 1

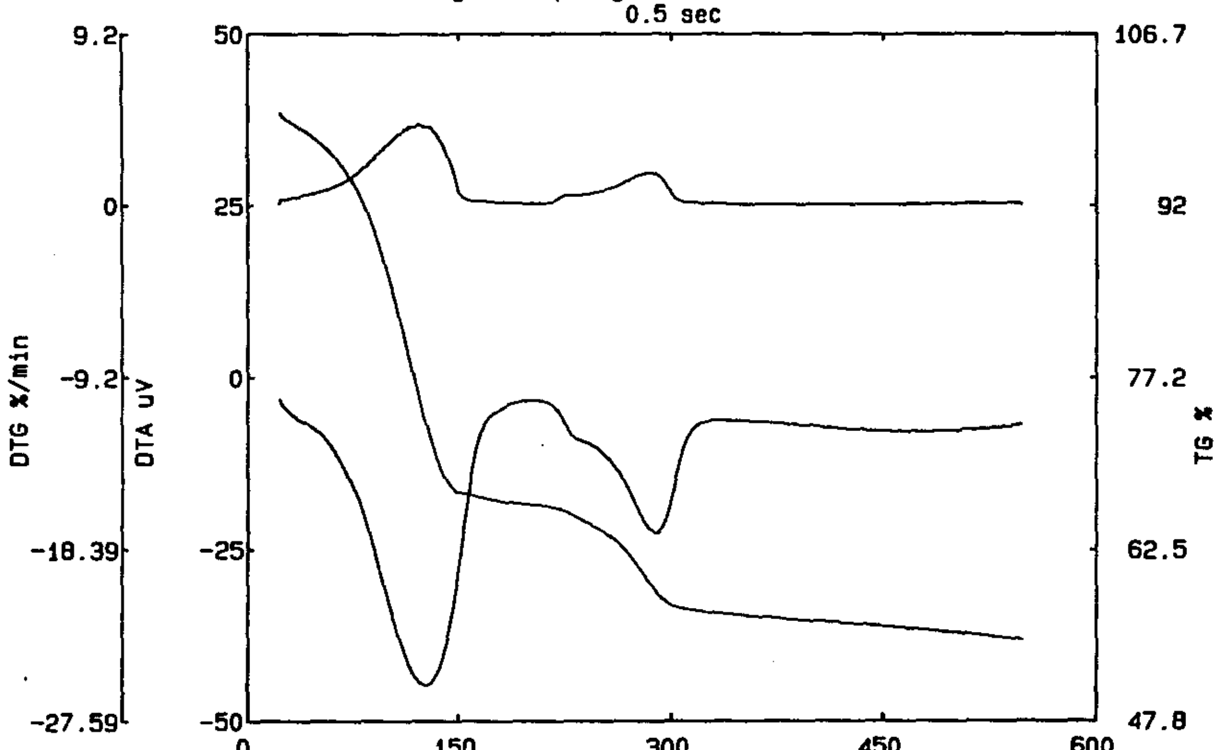
TG/DTA

<Name>
20sept95d
<Date>
95/09/20 08:41

<Sample>
20sept95d
27.185 mg
(27.185 mg)
<Reference>
pt pan
0.000 mg

<Comment>
7313
<Sampling>
0.5 sec

<Temp.program(C) (C/min) (min)>
1* 20.0- 550.0 10.00 0.00
<Gas>
nitrogen 300.0 ml/min
0.0 ml/min



Chemical Process Systems

3-230

WHC-SD-WM-DP-145, REV. 1

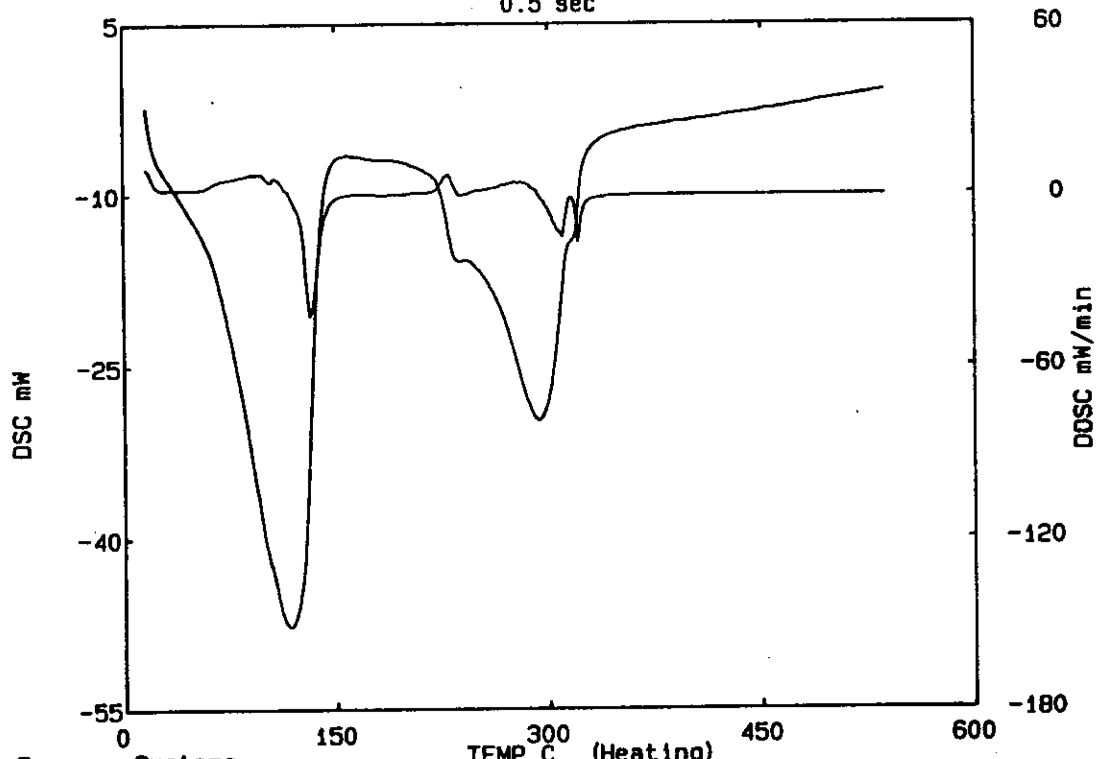
IMM

DSC
<Name>
20sept95e
<Date>
95/09/20 10:14

<Sample>
20sept95e
29.313 mg
(29.313 mg)
<Reference>
pt pan
0.000 mg

<Comment>
7313-2
<Sampling>
0.5 sec

<Temp.program [C] [C/min] [min]>
1* 20.0- 550.0 10.00 0.00
<Gas>
nitrogen 300.0 ml/min
0.0 ml/min



Chemical Process Systems

3-231

WHC-SD-WM-DP-145, REV.1

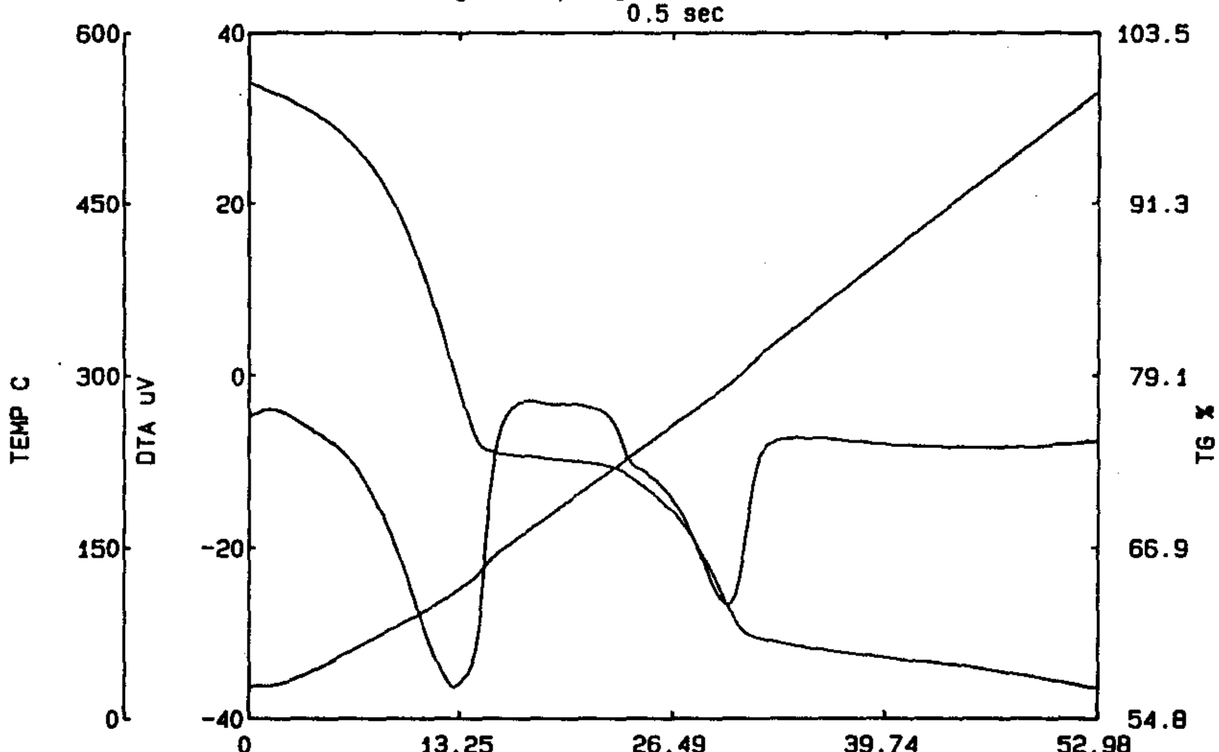
TG/DTA

<Name>
20sept95f
<Date>
95/09/20 10:18

<Sample>
20sept95f
24.648 mg
(24.648 mg)
<Reference>
pt pan
0.000 mg

<Comment>
7313-2
<Sampling>
0.5 sec

<Temp.program [C] [C/min] [min]>
1* 20.0- 550.0 10.00 0.00
<Gas>
nitrogen 300.0 ml/min
0.0 ml/min



Chemical Process Systems

WHC-SD-WM-DP-145, REV. 1

3-233

WHC-SD-WM-DP-145, REV. 1

DSC

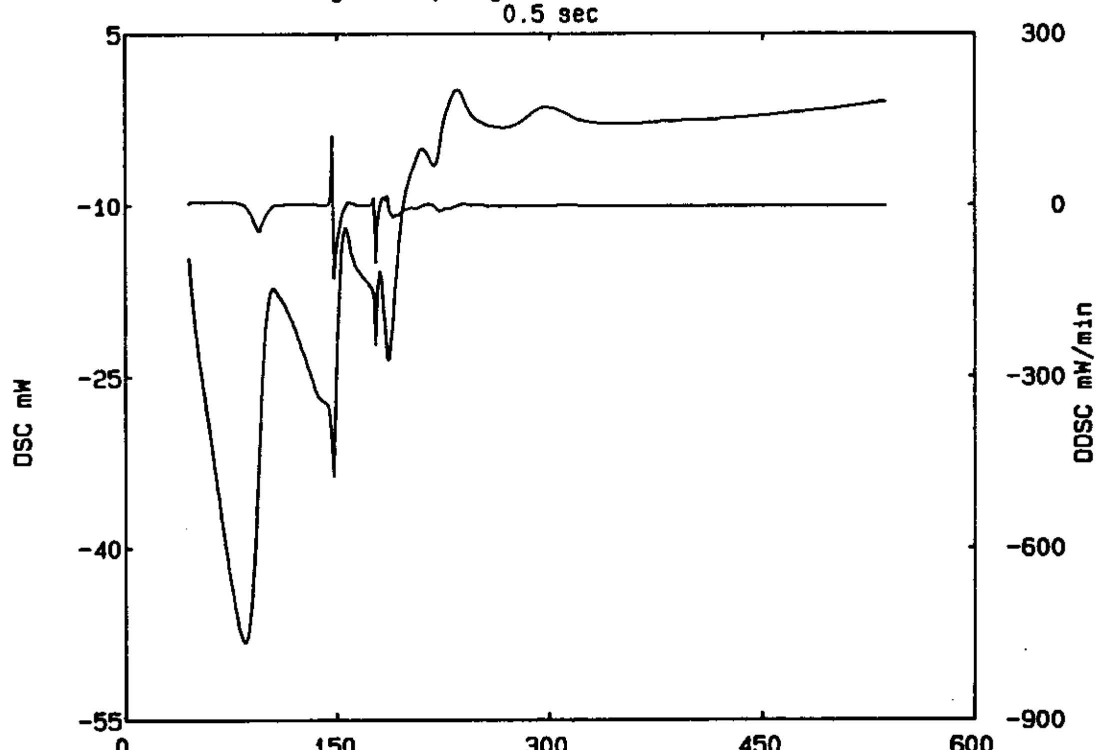
<Name>
19sept95k
<Date>
95/09/19 18:29

<Sample>
19sept95k
20.316 mg
(20.316 mg)
<Reference>
pt pan
0.000 mg

<Comment>
95-07932

<Sampling>
0.5 sec

<Temp.program(C) (C/min) (min)>
1* 20.0- 550.0 10.00 0.00
<Gas>
nitrogen 300.0 ml/min
--------------- 0.0 ml/min



Chemical Process Systems

1080

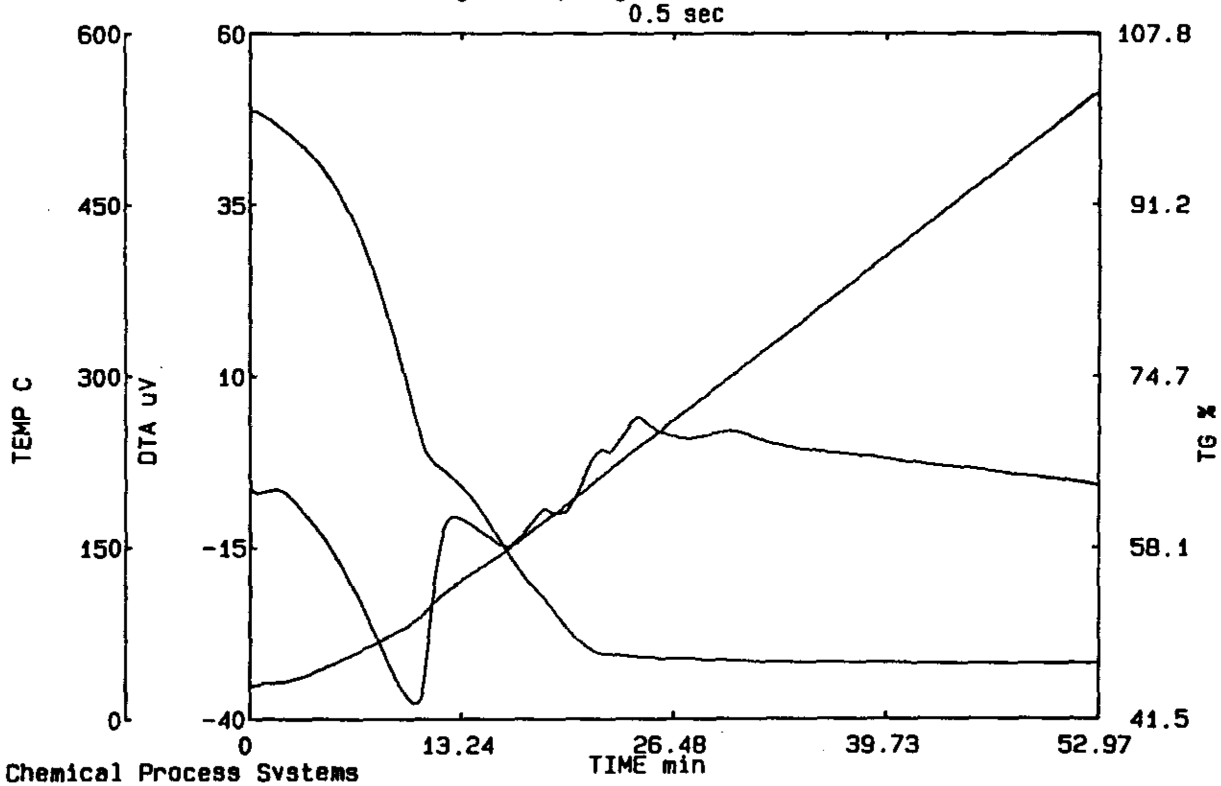
3-234

TG/DTA
<Name>
19sept951
<Date>
95/09/19 18:32

<Sample>
19septy951
18.083 mg
(18.083 mg)
<Reference>
pt pan
0.000 mg

<Comment>
95-07932
<Sampling>
0.5 sec

<Temp.program[C] [C/min] [min]>
1* 20.0- 550.0 10.00 0.00
<Gas>
nitrogen 300.0 ml/min
0.0 ml/min



Chemical Process Systems

WHC-SD-WM-DP-145, REV. 1

3-235

DSC

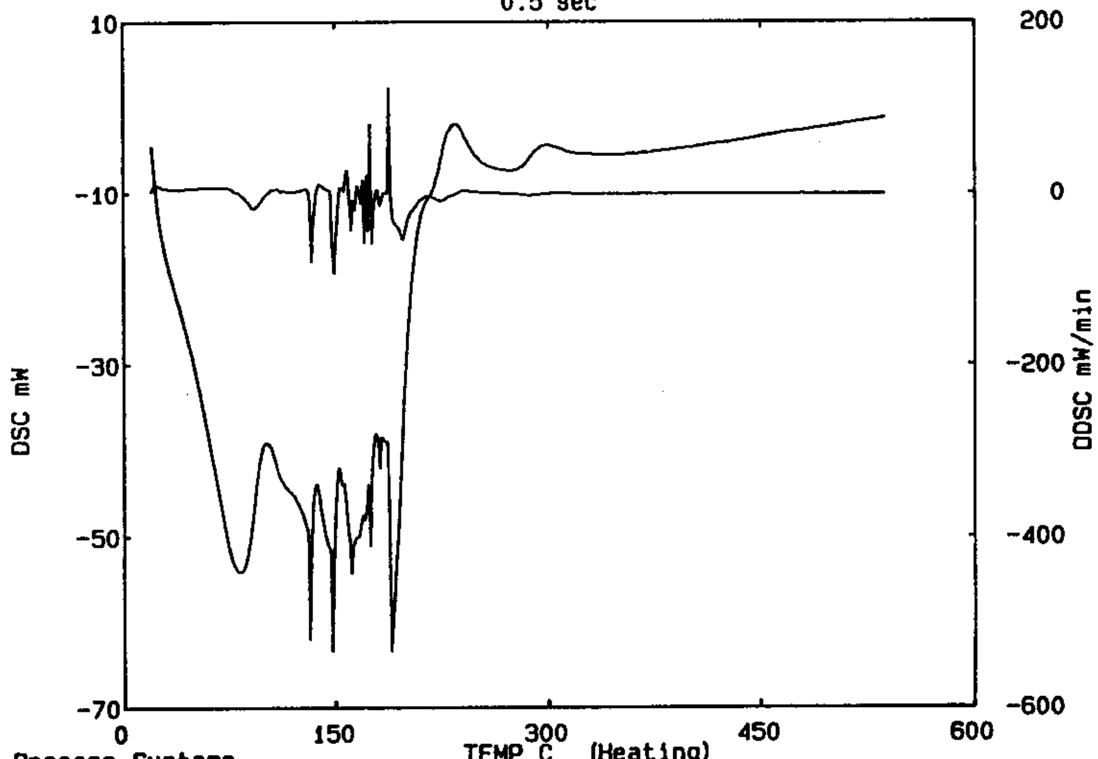
<Name>
20sept95a
<Date>
95/09/20 06:31

<Sample>
20sept95a
30.332 mg
(30.332 mg)
<Reference>
pt pan
0.000 mg

<Comment>
95-07932-2

<Sampling>
0.5 sec

<Temp.program [C] [C/min] [min]>
1* 20.0- 550.0 10.00 0.00
<Gas>
nitrogen 300.0 ml/min
--------------- 0.0 ml/min



Chemical Process Systems

WHC-SD-WM-DP-145, REV. 1

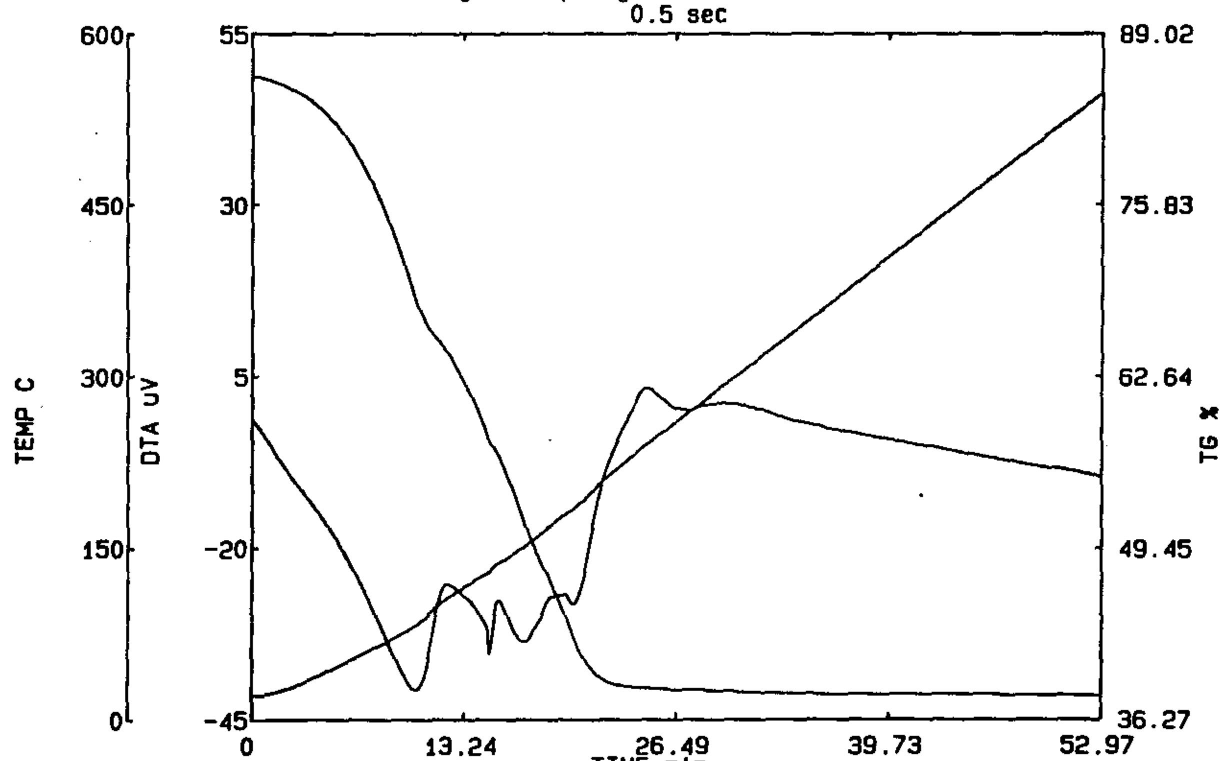
TG/DTA

<Name>
20sept95b
<Date>
95/09/20 06:32

<Sample>
20sept95b
30.332 mg
(30.332 mg)
<Reference>
pt pan
0.000 mg

<Comment>
95-07932-2
<Sampling>
0.5 sec

<Temp.program [C] [C/min] [min]>
1* 20.0- 550.0 10.00 0.00
<Gas>
nitrogen 300.0 ml/min
0.0 ml/min



Chemical Process Systems

TG/DTA

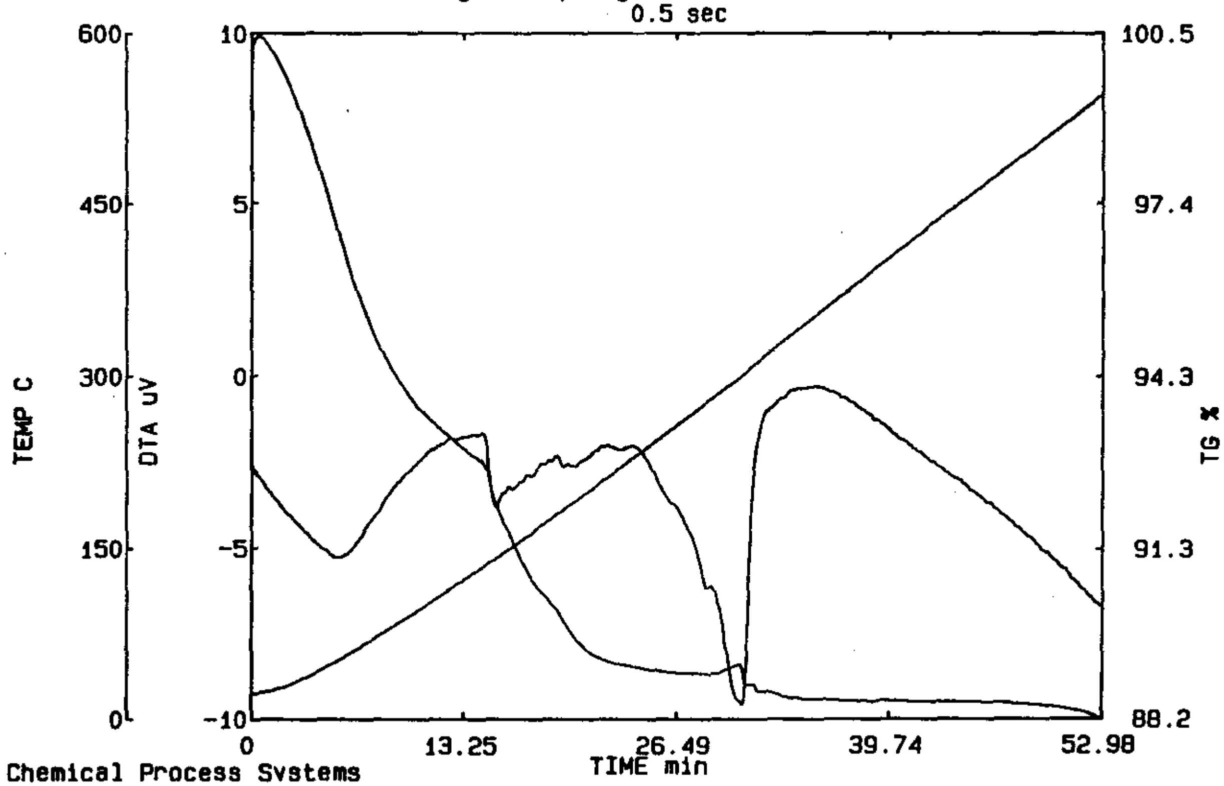
<Name>
21sept95f
<Date>
95/09/21 15:07

<Sample>
21sept95f
12.984 mg
(12.984 mg)
<Reference>
pt pan
0.000 mg

<Comment>
7315

<Sampling>
0.5 sec

<Temp.program [C] [C/min] [min]>
1* 20.0- 550.0 10.00 0.00
<Gas>
nitrogen 300.0 ml/min
--------------- 0.0 ml/min



Chemical Process Systems

3-237

WHC-SD-WM-DP-145, REV. 1

3- 238

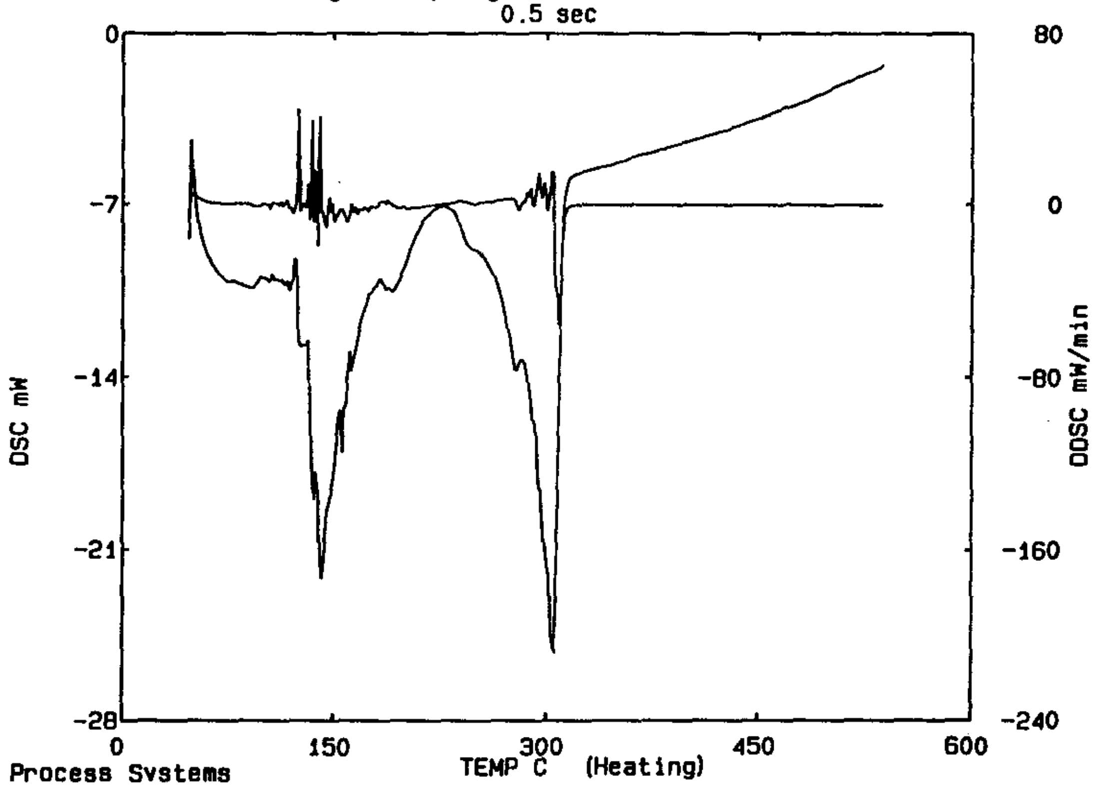
DSC

<Name>
21sept95e
<Date>
95/09/21 15:05

<Sample>
21sept95e
28.860 mg
(28.860 mg)
<Reference>
pt pan
0.000 mg

<Comment>
7315
<Sampling>
0.5 sec

<Temp.program [C] [C/min] [min]>
1* 20.0- 550.0 10.00 0.00
<Gas>
nitrogen 300.0 ml/min
0.0 ml/min



Chemical Process Systems

WHC-SD-WM-DP-145, REV 1

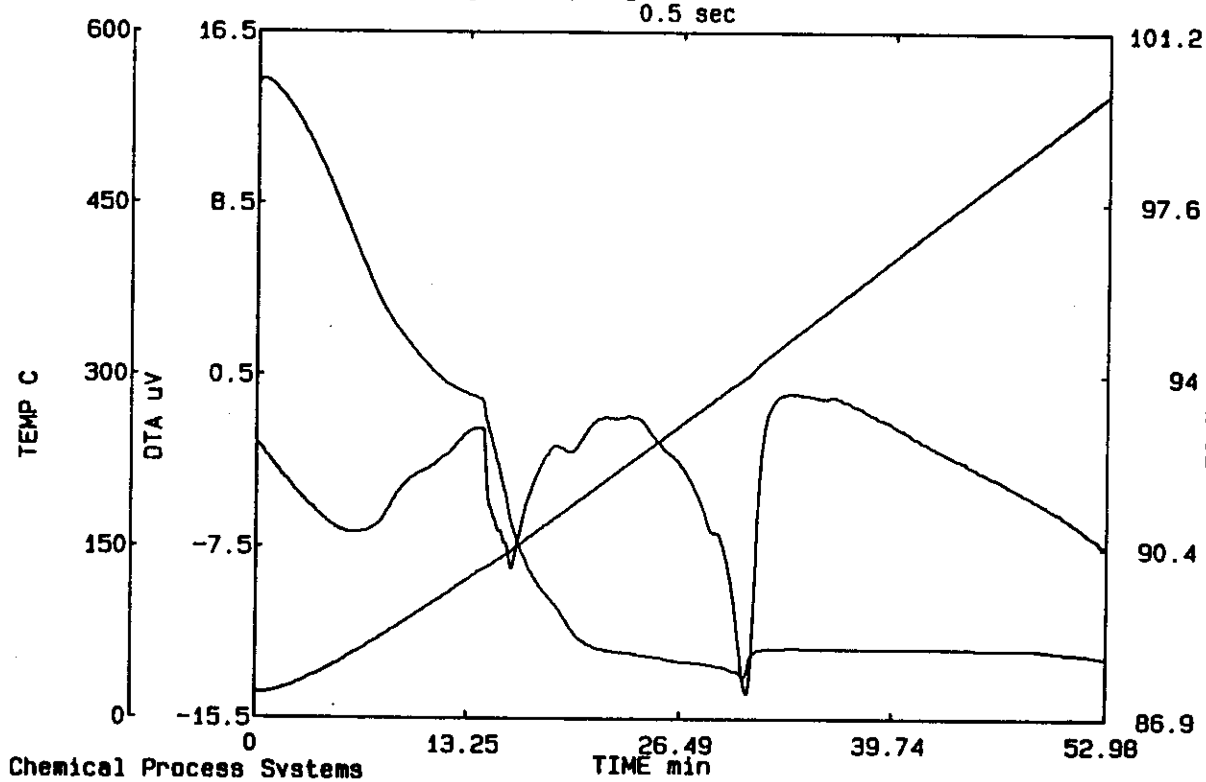
3-239

TG/DTA
<Name>
21sept95h
<Date>
95/09/21 16:57

<Sample>
21sept95h
22.391 mg
(22.391 mg)
<Reference>
pt pan
0.000 mg

<Comment>
7315-2
<Sampling>
0.5 sec

<Temp.program[C] [C/min] [min]>
1* 20.0- 550.0 10.00 0.00
<Gas>
nitrogen 300.0 ml/min
0.0 ml/min



WHC-SD-WM-DP-165, REV. 1

3-240

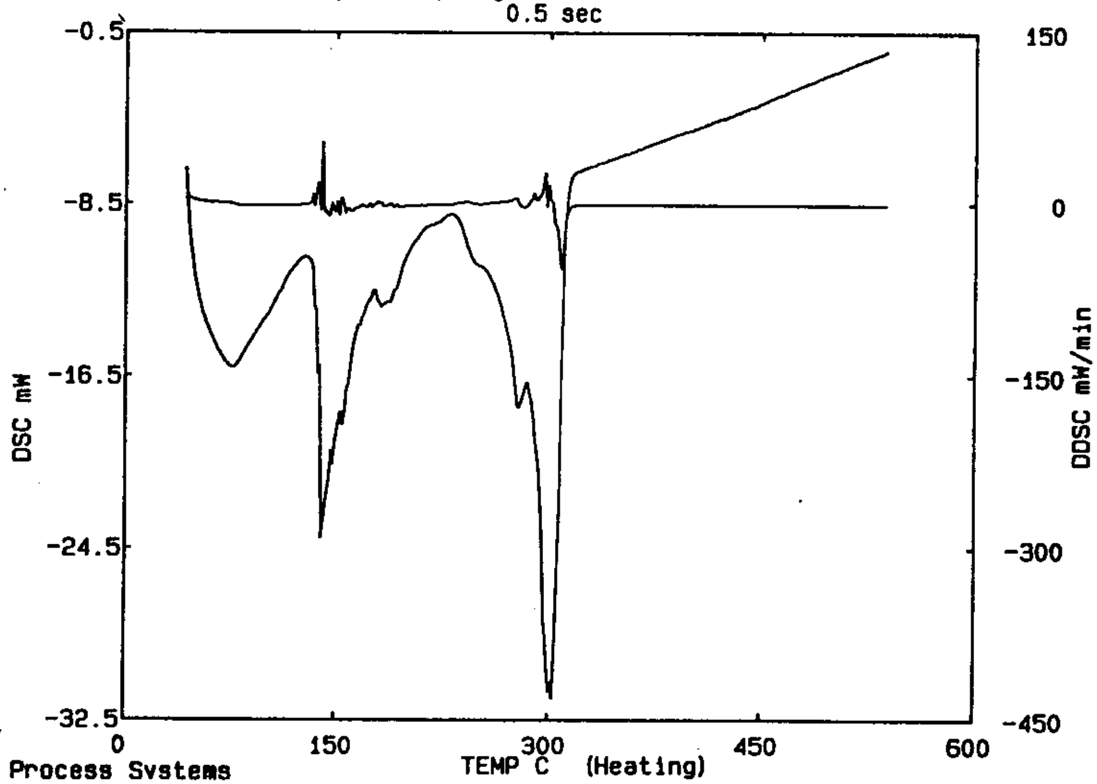
DSC

<Name>
21sept95g
<Date>
95/09/21 16:55

<Sample>
21sept95g
35.068 mg
(35.068 mg)
<Reference>
pt pan
0.000 mg

<Comment>
7315-2
<Sampling>
0.5 sec

<Temp.program [C] [C/min] [min]>
1* 20.0- 550.0 10.00 0.00
<Gas>
nitrogen 300.0 ml/min
0.0 ml/min



Chemical Process Systems

WHC-SD-WM-DP-145, REV. 1

DSC

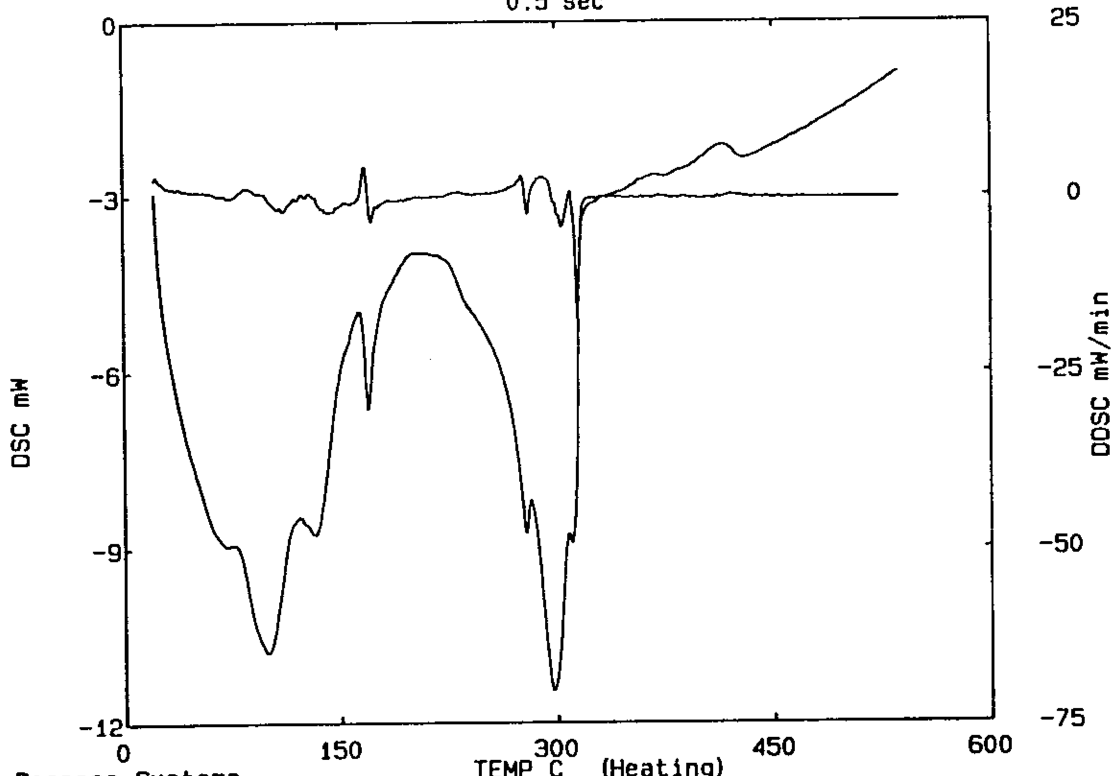
<Name>
21sept95a
<Date>
95/09/21 09:00

<Sample>
21sept95a
16.036 mg
(16.036 mg)
<Reference>
pt pan
0.000 mg

<Comment>
7314

<Sampling>
0.5 sec

<Temp.program[C] [C/min] [min]>
1* 20.0- 550.0 10.00 0.00
<Gas>
nitrogen 300.0 ml/min
0.0 ml/min



Chemical Process Systems

3- 241

WHC-SD-WM-DP-145, REV. 1

3-242

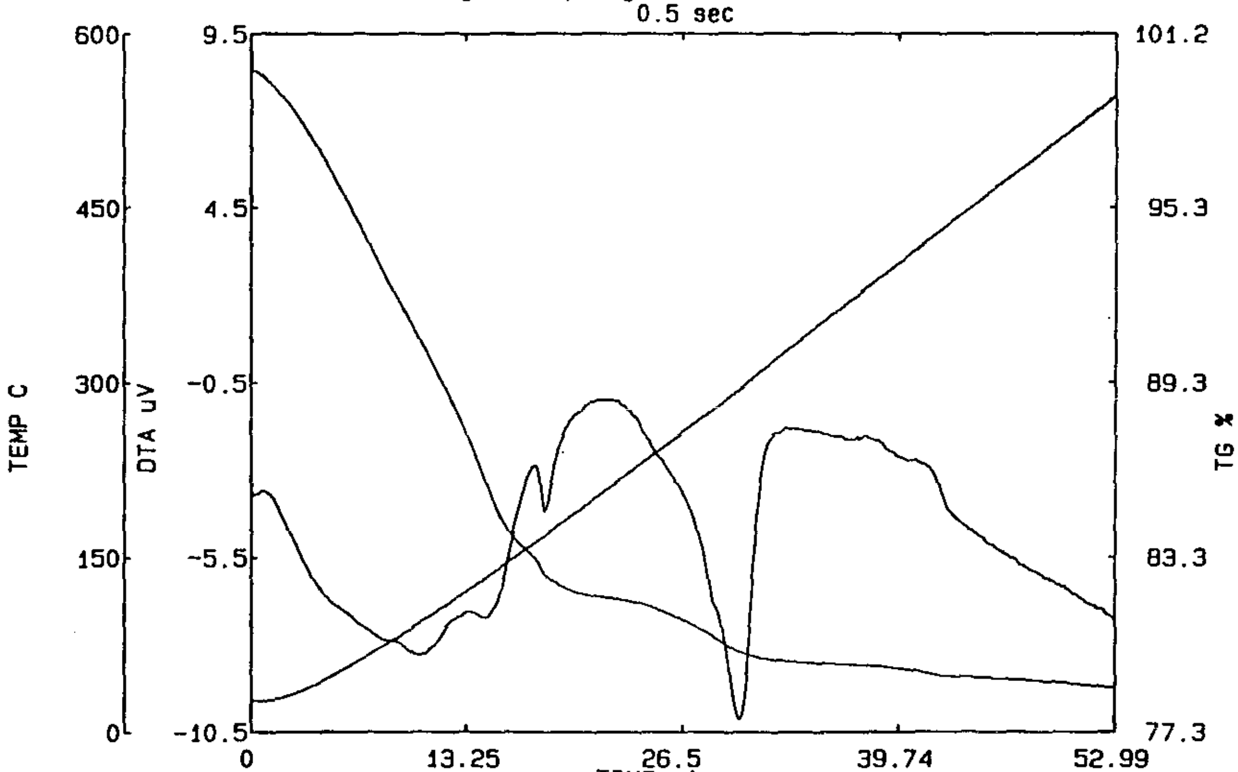
TG/DTA

<Name>
21sept95b
<Date>
95/09/21 09:04

<Sample>
21sept95b
15.063 mg
(15.063 mg)
<Reference>
pt pan
0.000 mg

<Comment>
7314
<Sampling>
0.5 sec

<Temp.program [C] [C/min] [min]>
1* 20.0- 550.0 10.00 0.00
<Gas>
nitrogen 300.0 ml/min
0.0 ml/min



Chemical Process Systems

WHC-SD-WM-DP-145, REV. 1

1A10

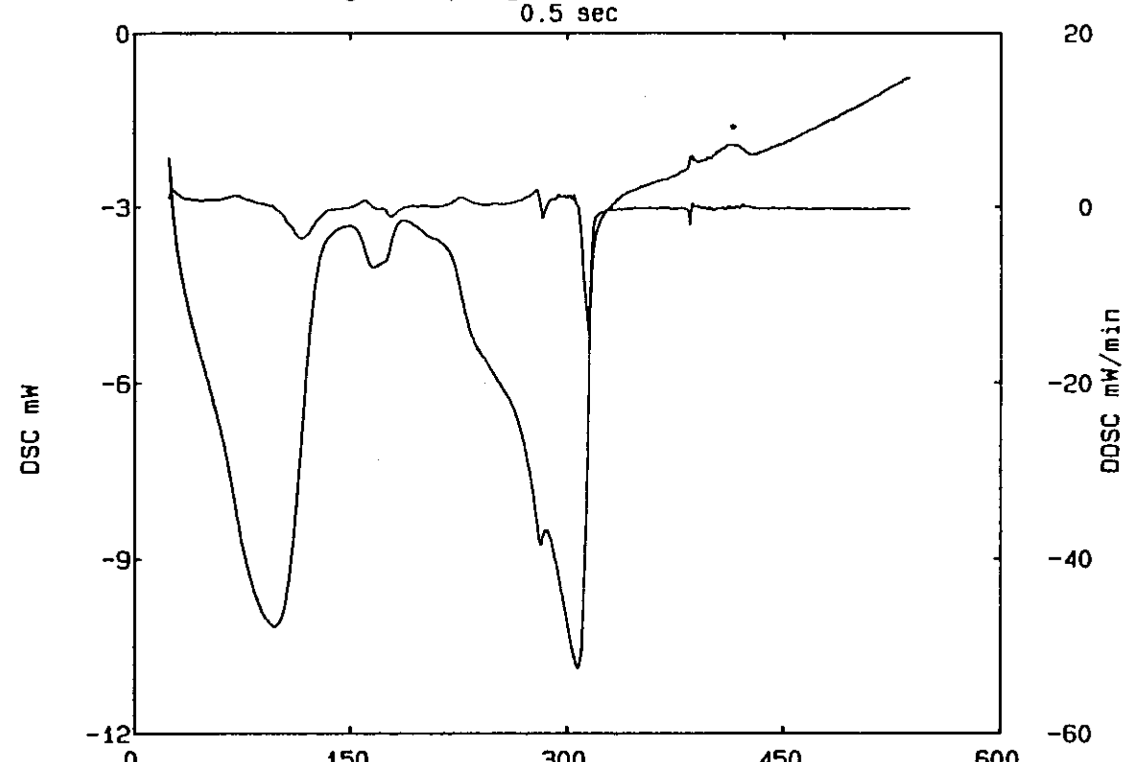
DSC

<Name>
21sept95c
<Date>
95/09/21 13:34

<Sample>
21sept95c
16.004 mg
(16.004 mg)
<Reference>
pt pan
0.000 mg

<Comment>
7314-2
<Sampling>
0.5 sec

<Temp.program(C) (C/min) (min)>
1* 20.0- 550.0 10.00 0.00
<Gas>
nitrogen 300.0 ml/min
0.0 ml/min



Chemical Process Systems

TEMP C (Heating)

3-243

WHC-SD-WM-DP-145, REV 1

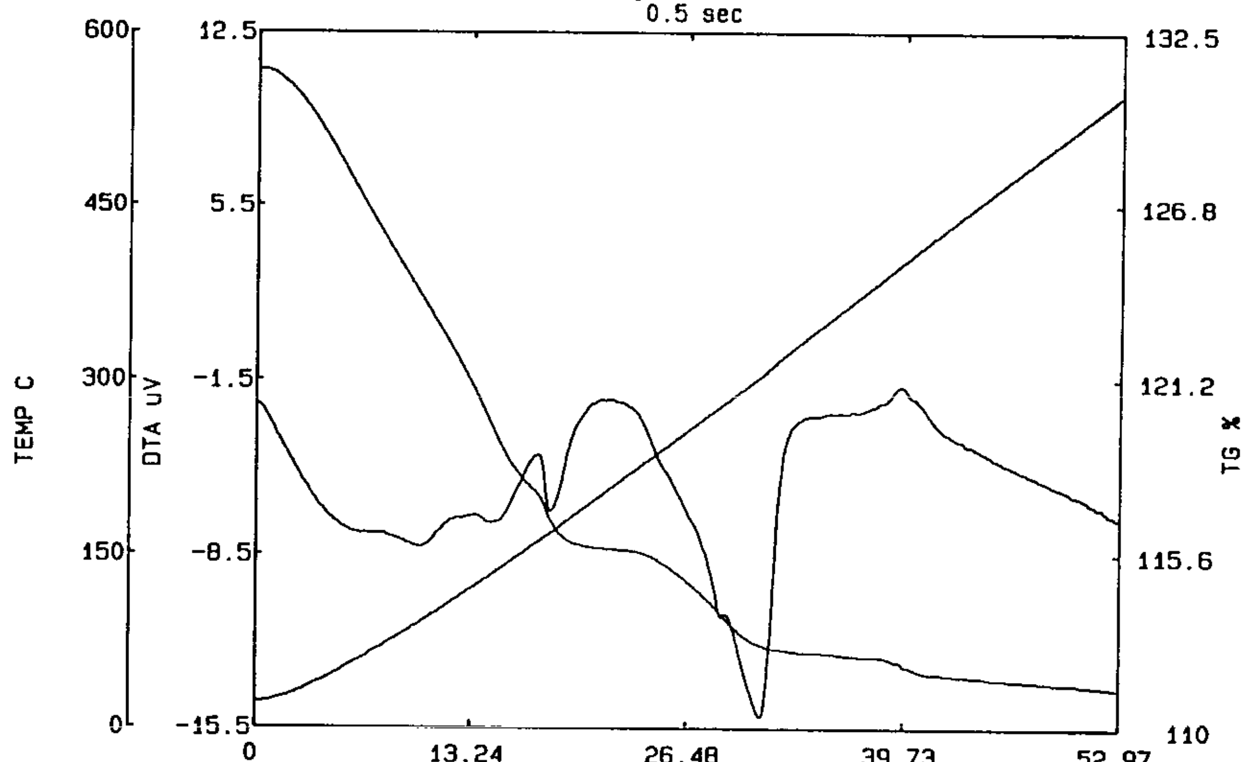
3-244

TG/DTA
<Name>
21sept95d
<Date>
95/09/21 13:34

<Sample>
21sept95d
16.004 mg
(16.004 mg)
<Reference>
pt pan
0.000 mg

<Comment>
7314-2
<Sampling>
0.5 sec

<Temp.program[C] [C/min] [min]>
1* 20.0- 550.0 10.00 0.00
<Gas>
nitrogen 300.0 ml/min
0.0 ml/min



Chemical Process Systems

WHC-SD-WM-DP-145, REV. 1

DSC

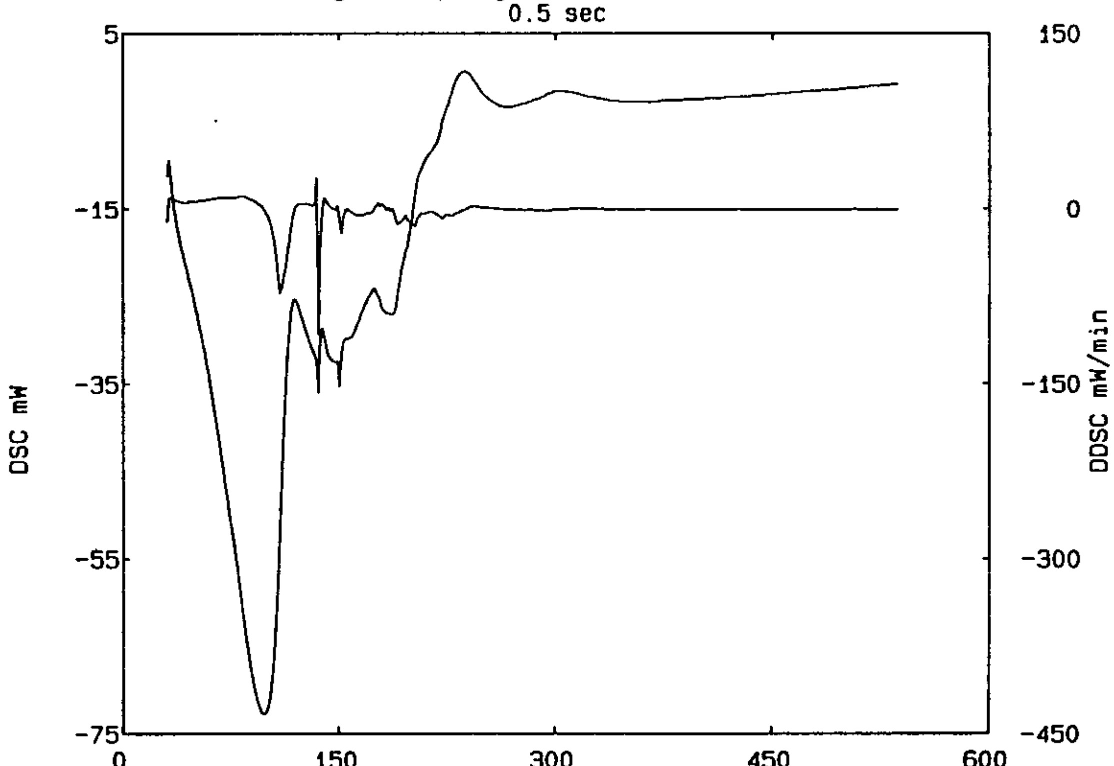
<Name>
19sept95g
<Date>
95/09/19 14:06

<Sample>
19sept95g
26.977 mg
(26.977 mg)
<Reference>
pt pan
0.000 mg

<Comment>
95-07935

<Sampling>
0.5 sec

<Temp.program [C] [C/min] [min]>
1* 20.0- 550.0 10.00 0.00
<Gas>
nitrogen 300.0 ml/min
0.0 ml/min



Chemical Process Systems

3-245

...-DF-145, REV. 1

3-246

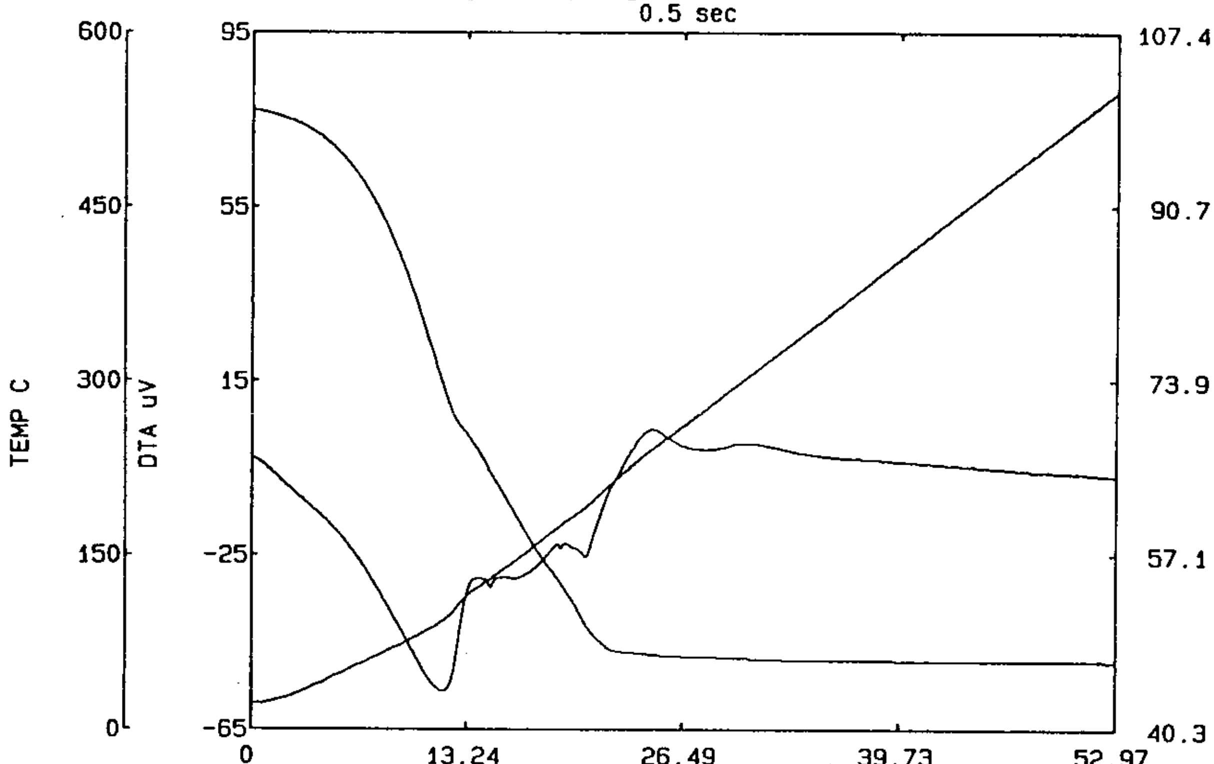
TG/DTA

<Name>
19sept95h
<Date>
95/09/19 14:09

<Sample>
19sept95h
29.784 mg
(29.784 mg)
<Reference>
pt pan
0.000 mg

<Comment>
95-07935
<Sampling>
0.5 sec

<Temp.program(C) (C/min) (min)>
1* 20.0- 550.0 10.00 0.00
<Gas>
nitrogen 300.0 ml/min
0.0 ml/min



Chemical Process Systems

WHC-SD-WM-DP-145, REV 1

3-247

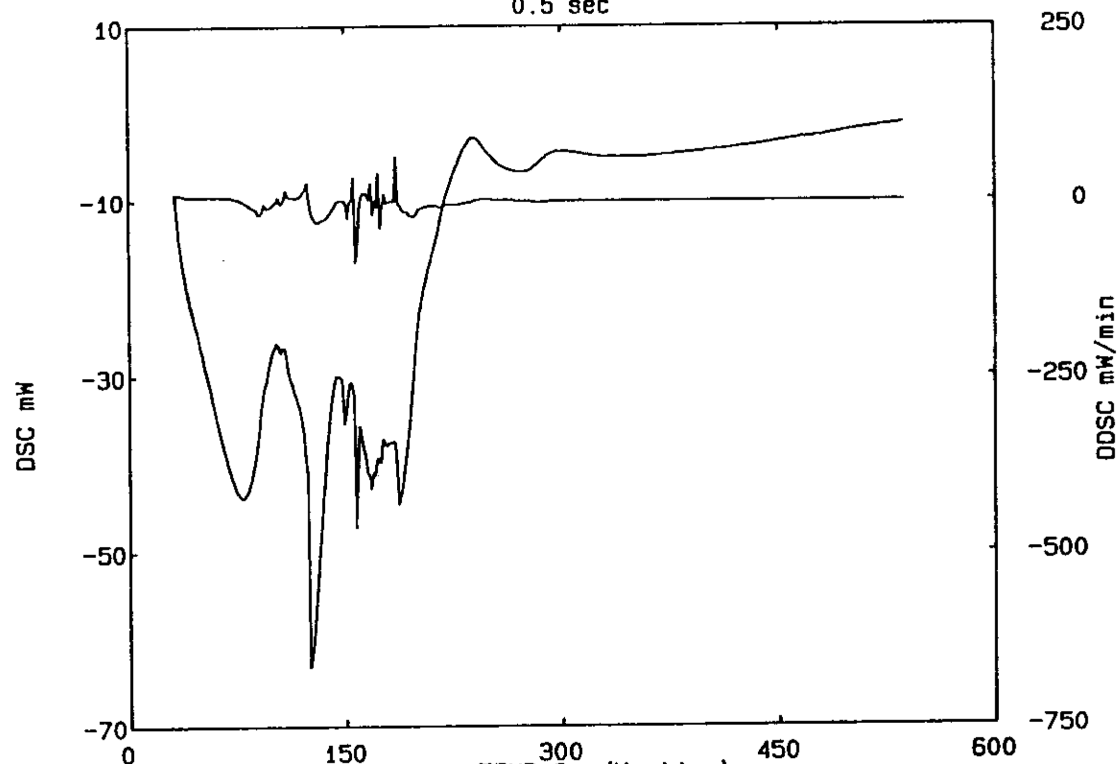
DSC

<Name>
19sept95i
<Date>
95/09/19 16:58

<Sample>
19sept95i
24.205 mg
(24.205 mg)
<Reference>
pt pan
0.000 mg

<Comment>
95-07935-2
<Sampling>
0.5 sec

<Temp.program [C] [C/min] [min]>
1* 20.0- 550.0 10.00 0.00
<Gas>
nitrogen 300.0 ml/min
0.0 ml/min



Chemical Process Systems

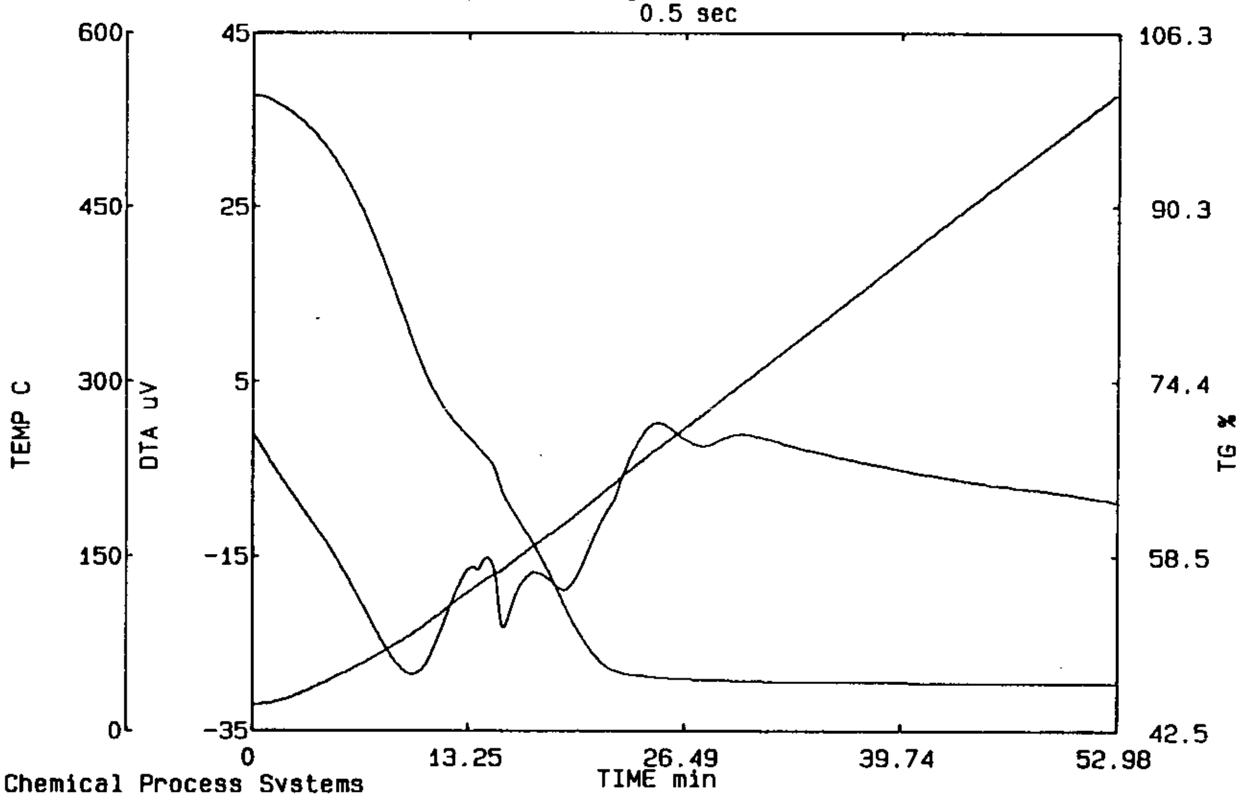
WHC-SD-WM-DP-165, REV 1

TG/DTA
<Name>
19sept95j
<Date>
95/09/19 17:01

<Sample>
19sept95j
18.818 mg
(18.818 mg)
<Reference>
pt pan
0.000 mg

<Comment>
95-07935-2
<Sampling>
0.5 sec

<Temp.program [C] [C/min] [min]>
1* 20.0- 550.0 10.00 0.00
<Gas>
nitrogen 300.0 ml/min
0.0 ml/min



Chemical Process Systems

3-248

WHC-SD-WM-DP-145, REV. 1

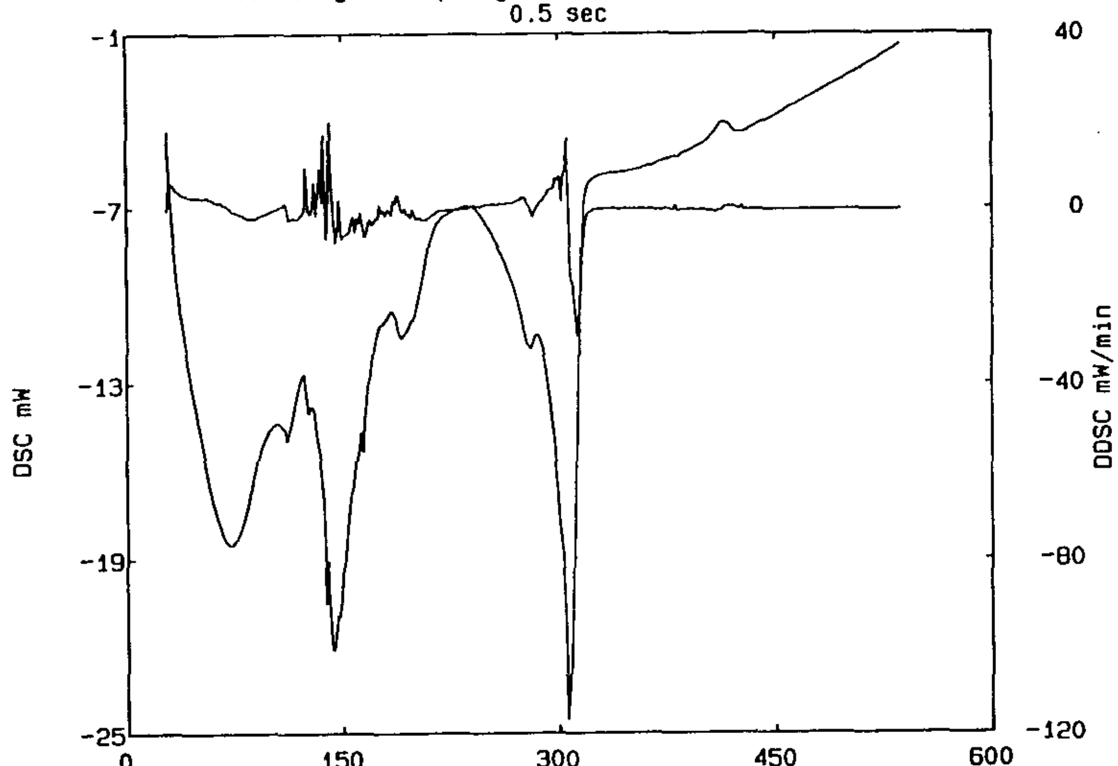
DSC

<Name>
20sept95g
<Date>
95/09/20 12:26

<Sample>
20sept95g
31.843 mg
(31.843 mg)
<Reference>
pt pan
0.000 mg

<Comment>
7316
<Sampling>
0.5 sec

<Temp.program [C] [C/min] [min]>
1* 20.0- 550.0 10.00 0.00
<Gas>
nitrogen 300.0 ml/min
0.0 ml/min



Chemical Process Systems

WHC-SD-WM-DP-145, REV. 1

3-249

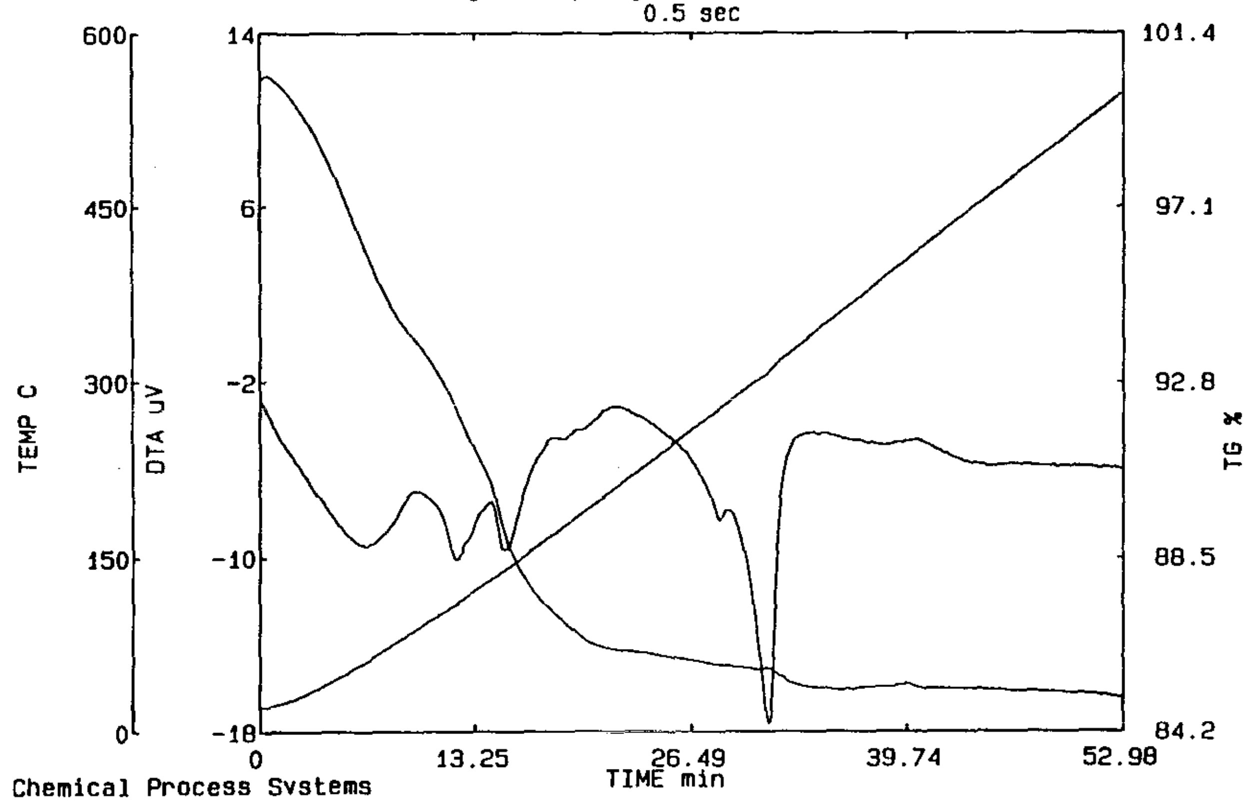
TG/DTA

<Name>
20sept95h
<Date>
95/09/20 12:29

<Sample>
20sept95h
23.331 mg
(23.331 mg)
<Reference>
pt pan
0.000 mg

<Comment>
7316
<Sampling>
0.5 sec

<Temp.program [C] [C/min] [min]>
1* 20.0~ 550.0 10.00 0.00
<Gas>
nitrogen 300.0 ml/min
0.0 ml/min



Chemical Process Systems

1020

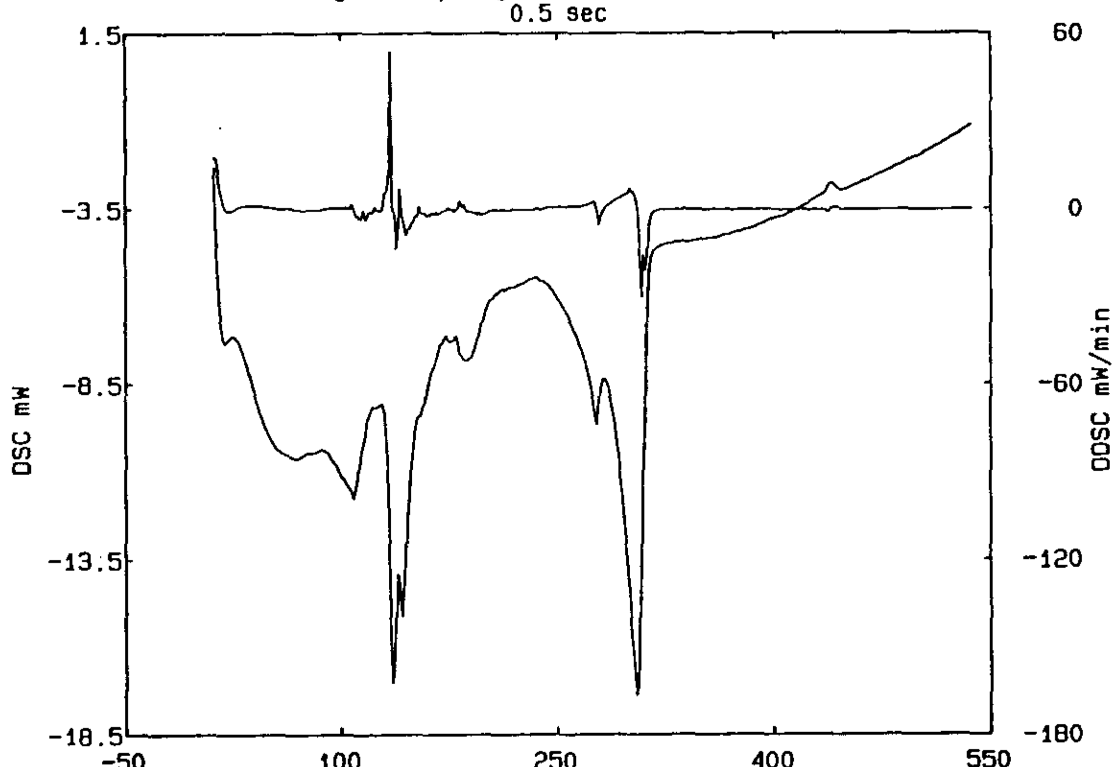
DSC

<Name>
20sept95i
<Date>
95/09/20 13:47

<Sample>
20sept95i
18.382 mg
(18.382 mg)
<Reference>
pt pan
0.000 mg

<Comment>
7316-2
<Sampling>
0.5 sec

<Temp.program[C] [C/min] [min]>
1* 20.0- 550.0 10.00 0.00
<Gas>
nitrogen 300.0 ml/min
0.0 ml/min



Chemical Process Systems

3-251

WHC-SD-WM-DP-145, REV.1

TG/DTA

<Name>
20sept95j

<Date>
95/09/20 13:49

<Sample>
20sept95j
18.382 mg
(18.382 mg)

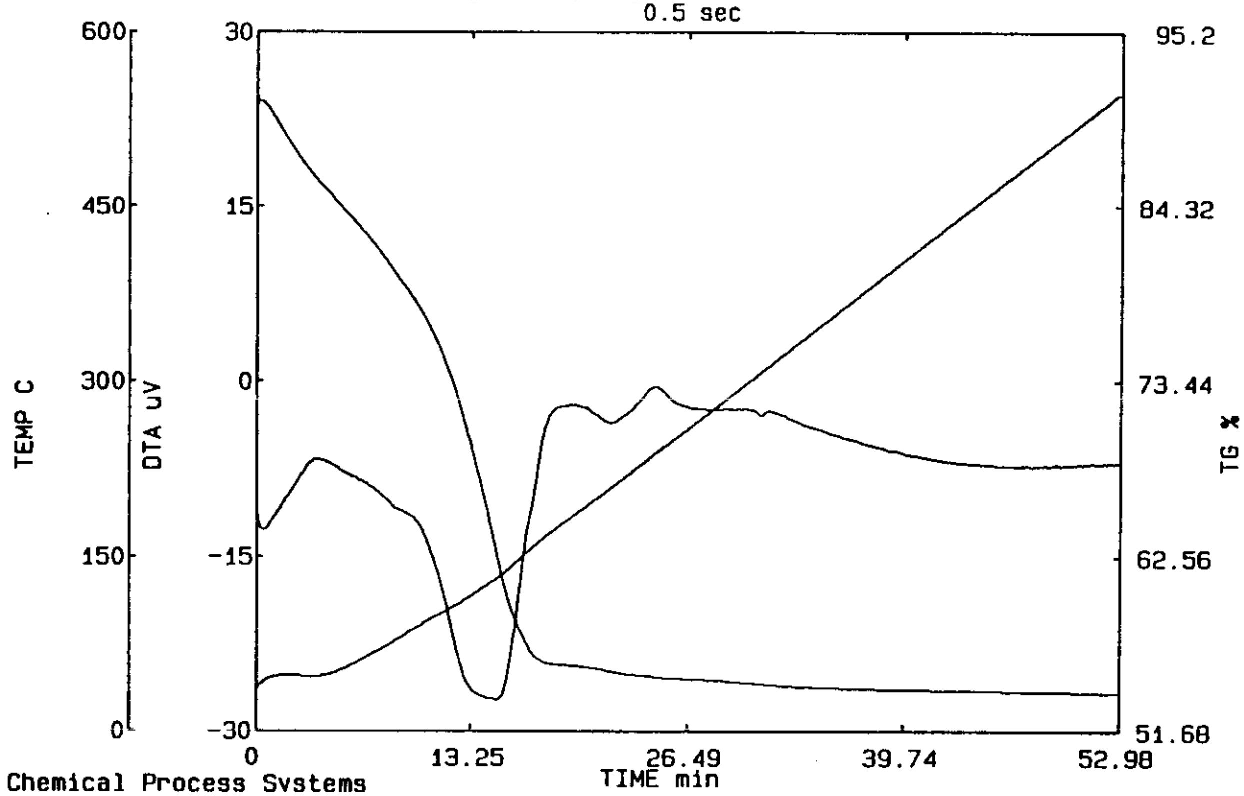
<Reference>
pt pan
0.000 mg

<Comment>
7316-2

<Sampling>
0.5 sec

<Temp.program [C] [C/min] [min]>
1* 20.0- 550.0 10.00 0.00

<Gas>
nitrogen 300.0 ml/min
0.0 ml/min



DSC

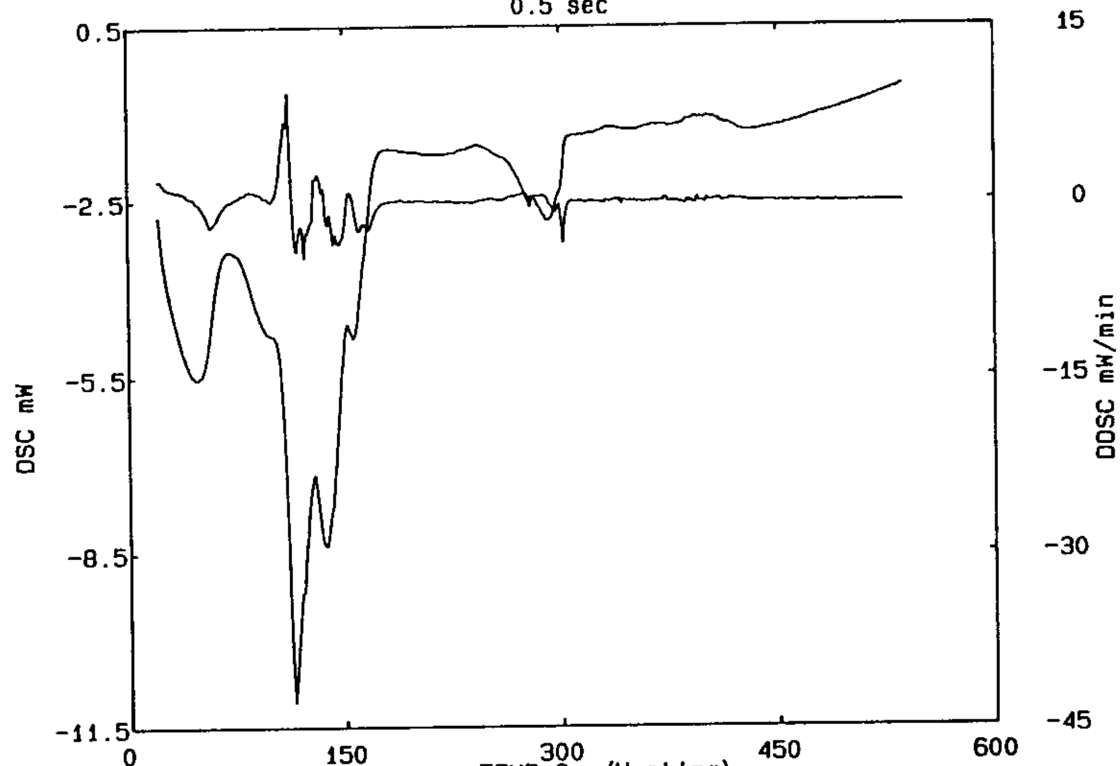
<Name>
15sept95a
<Date>
95/09/15 06:31

<Sample>
15sept95a
7.391 mg
(7.391 mg)
<Reference>
pt pan
0.000 mg

<Comment>
7322

<Sampling>
0.5 sec

<Temp.program[C] [C/min] [min]>
1* 20.0- 550.0 10.00 0.00
<Gas>
nitrogen 300.0 ml/min
0.0 ml/min



Chemical Process Systems

3-253

WHC-SD-WM-DP-145, REV. 1

TG/DTA

<Name> 15sept95b

<Date> 95/09/15 06:36

<Sample> 15sept95b 17.351 mg (17.351 mg)

<Reference> pt pan 0.000 mg

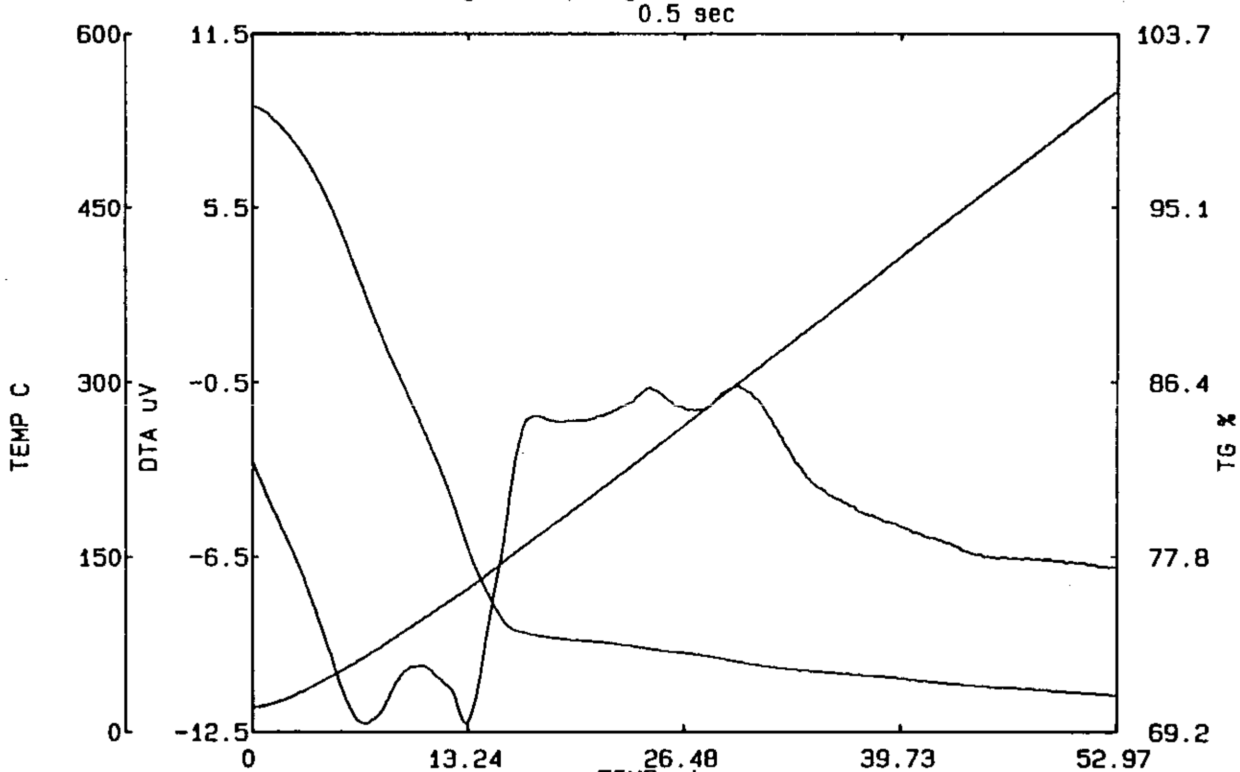
<Comment> 7322

<Sampling> 0.5 sec

<Temp.program [C] [C/min] [min]> 1* 20.0- 550.0 10.00 0.00

<Gas> nitrogen 300.0 ml/min

0.0 ml/min



Chemical Process Systems

3-254

WHC-SD-WM-DP-145, REV. 1

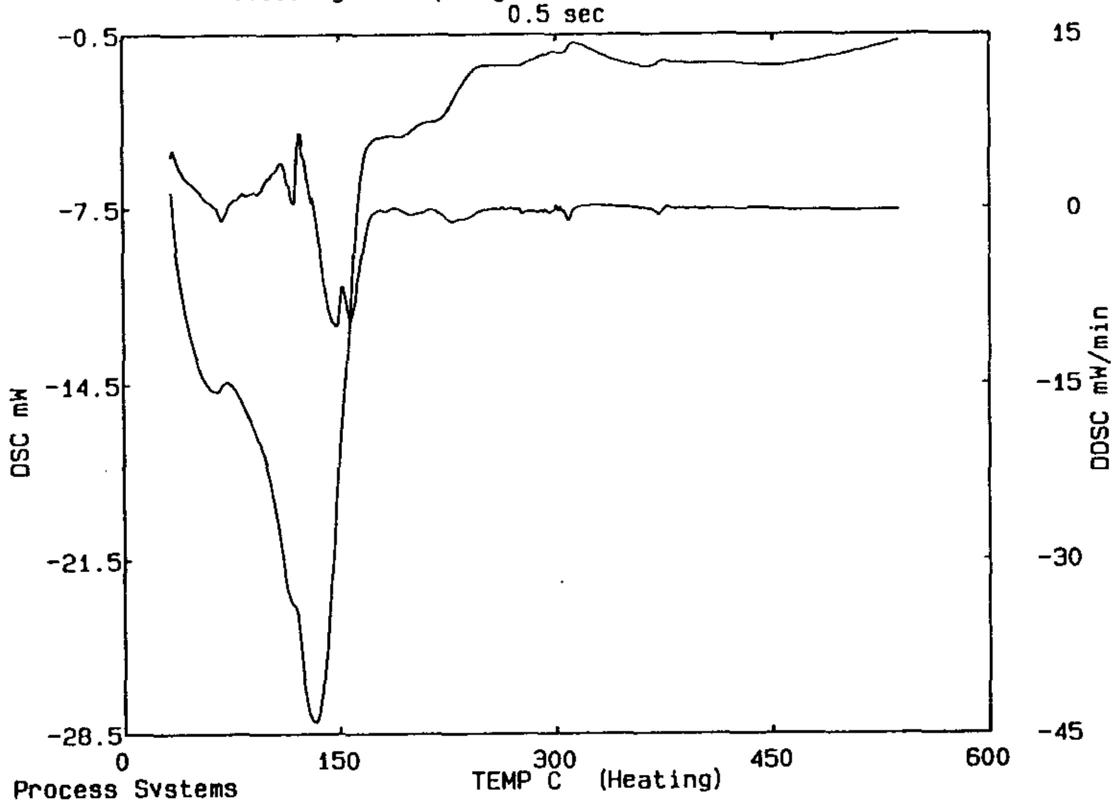
DSC

<Name>
19sept95e
<Date>
95/09/19 11:35

<Sample>
19sept95e
18.071 mg
(18.071 mg)
<Reference>
pt pan
0.000 mg

<Comment>
7322-2
<Sampling>
0.5 sec

<Temp.program [C] [C/min] [min]>
1* 20.0- 550.0 10.00 0.00
<Gas>
nitrogen 300.0 ml/min
0.0 ml/min



Chemical Process Systems

3-255

WHC-SD-WM-DP-145, REV. 1

3-256

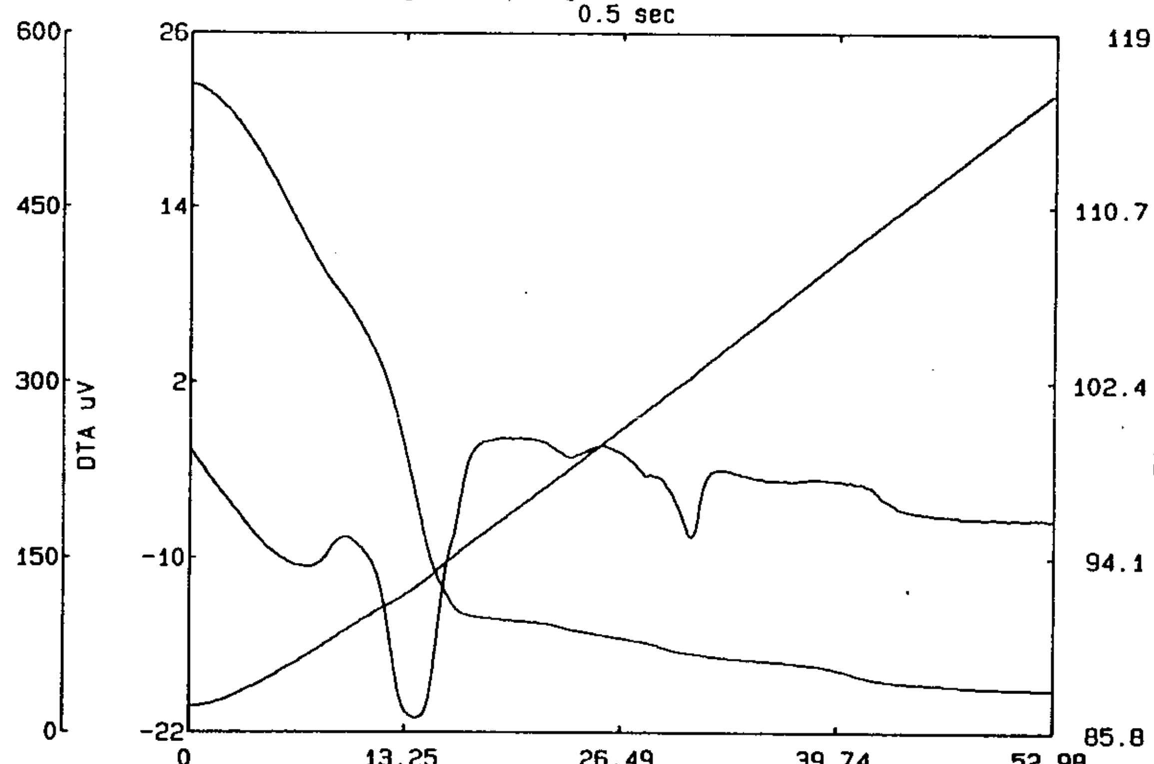
TG/DTA

<Name>
19sept95f
<Date>
95/09/19 11:38

<Sample>
19sept95f
18.071 mg
(18.071 mg)
<Reference>
pt pan
0.000 mg

<Comment>
7322-2
<Sampling>
0.5 sec

<Temp.program[C] [C/min] [min]>
1* 20.0- 550.0 10.00 0.00
<Gas>
nitrogen 300.0 ml/min
0.0 ml/min



Chemical Process Systems

WHC-SD-WM-DP-145, REV. 1

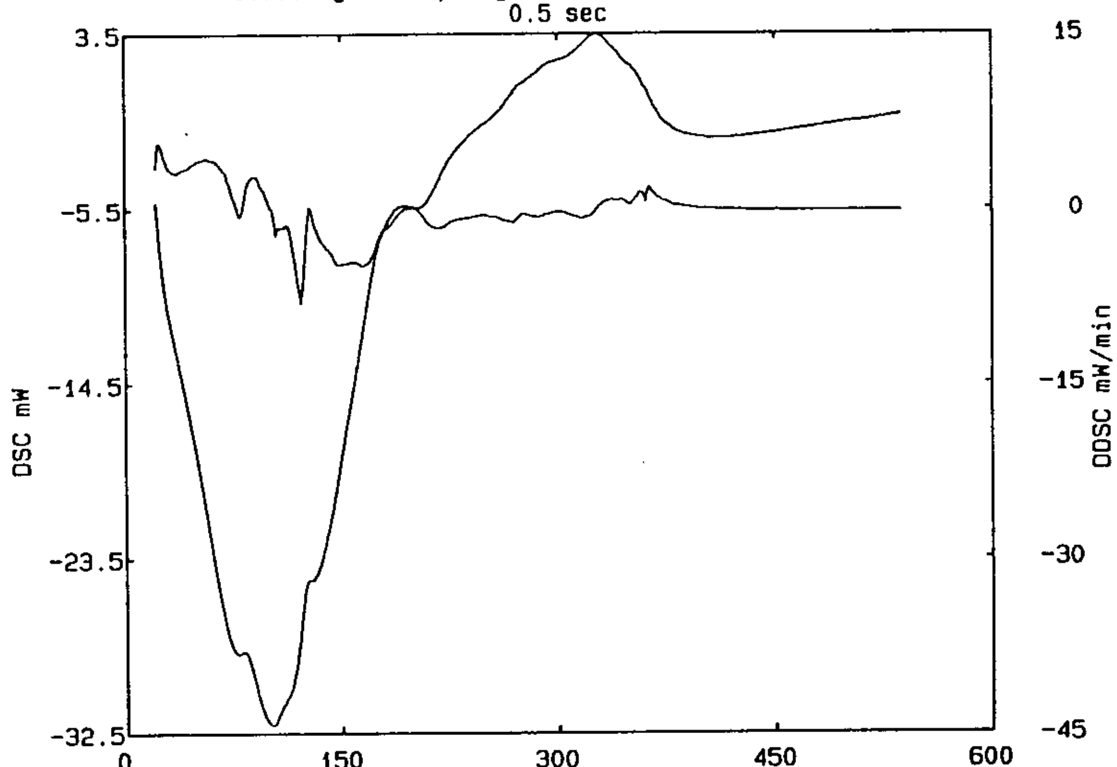
DSC

<Name>
19sept95a
<Date>
95/09/19 06:52

<Sample>
19sept95a
23.424 mg
(23.424 mg)
<Reference>
pt pan
0.000 mg

<Comment>
7319
<Sampling>
0.5 sec

<Temp.program [C] [C/min] [min]>
1* 20.0- 550.0 10.00 0.00
<Gas>
nitrogen 300.0 ml/min
0.0 ml/min



Chemical Process Systems

3-257

WHC-SD-WM-DP-145, REV. 1

TG/DTA

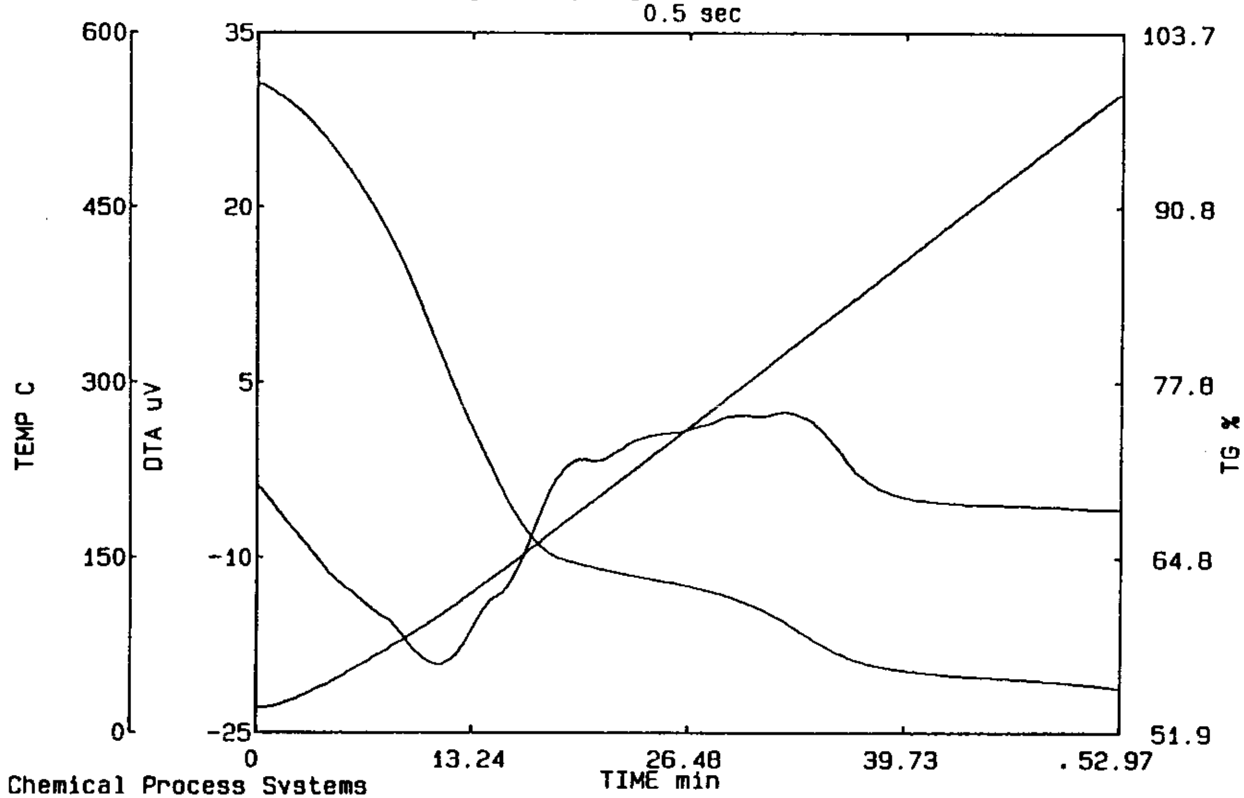
<Name>
19sept95b
<Date>
95/09/19 06:55

<Sample>
19sept95b
19.283 mg
(19.283 mg)
<Reference>
pt pan
0.000 mg

<Comment>
7319

<Sampling>
0.5 sec

<Temp.program [C] [C/min] [min]>
1* 20.0- 550.0 10.00 0.00
<Gas>
nitrogen 300.0 ml/min
0.0 ml/min



Chemical Process Systems

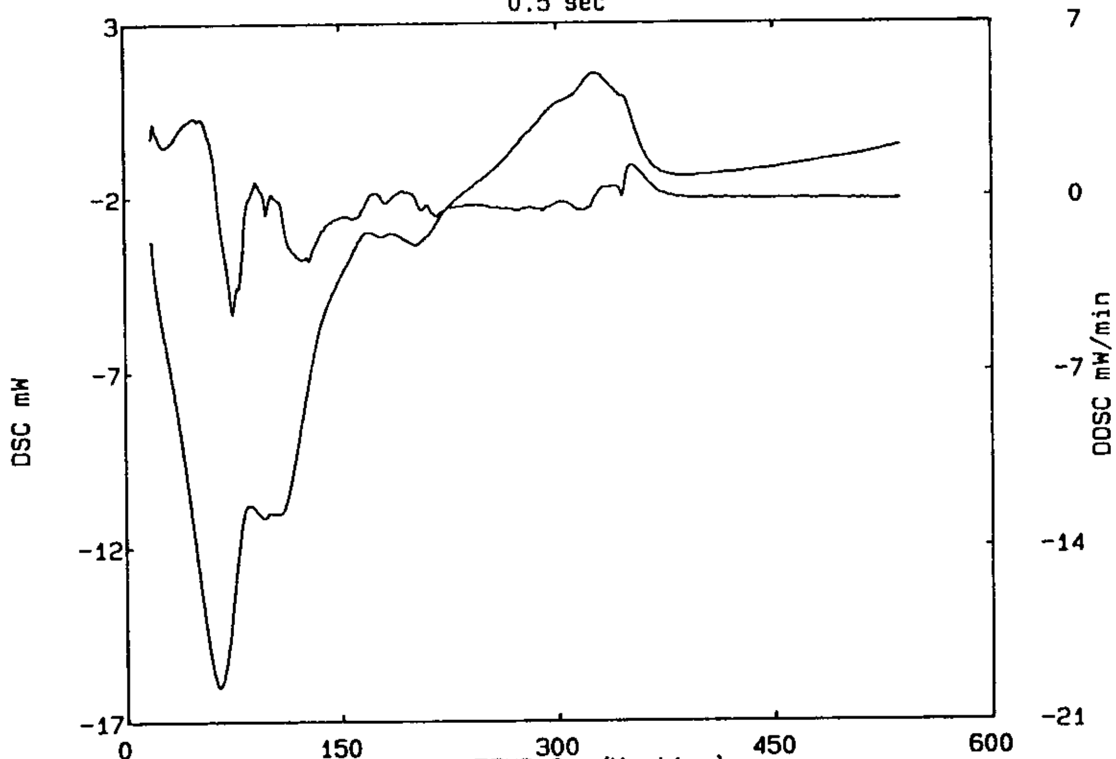
DSC

<Name>
19sept95c
<Date>
95/09/19 08:57

<Sample>
19sept95c
12.027 mg
(12.027 mg)
<Reference>
pt pan
0.000 mg

<Comment>
7319-2
<Sampling>
0.5 sec

<Temp.program[C] [C/min] [min]>
1* 20.0- 550.0 10.00 0.00
<Gas>
nitrogen 300.0 ml/min
0.0 ml/min



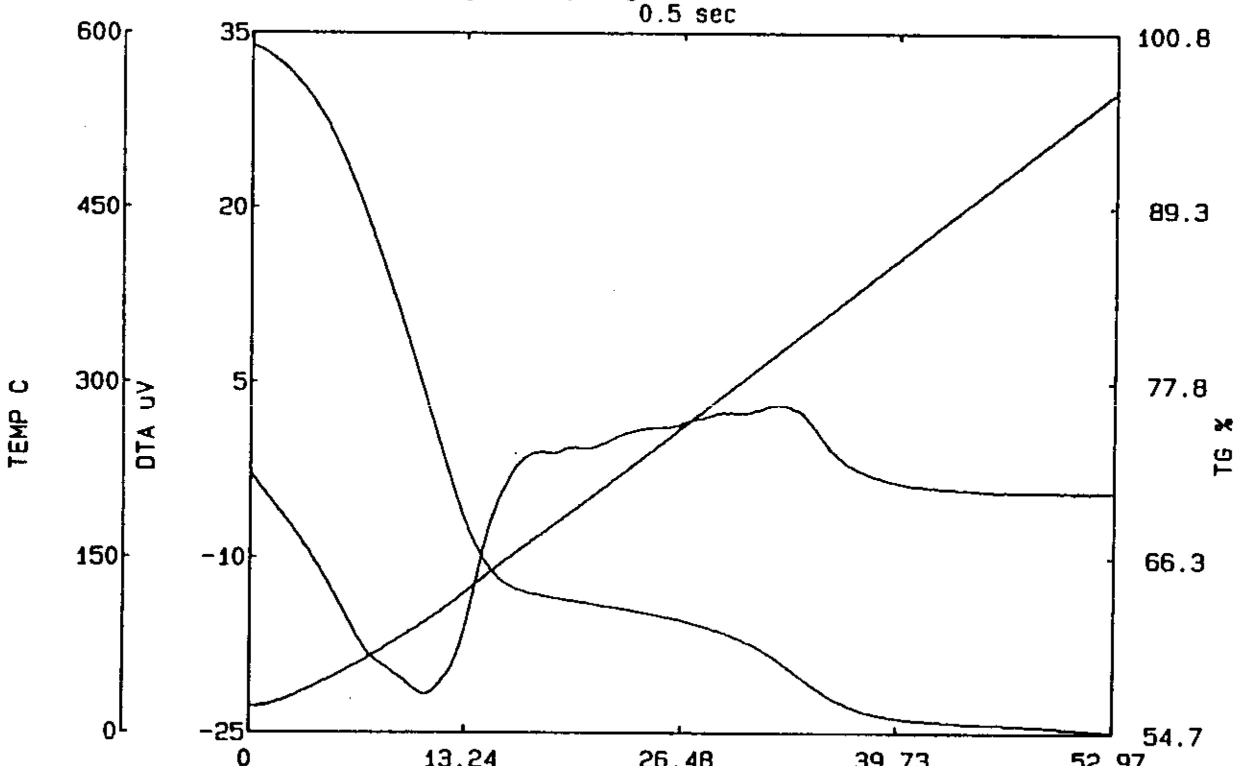
Chemical Process Systems

3-259

WHC-SD-WM-DP-145, REV 1

TG/DTA

<Name> 19sept95d	<Sample> 19sept95d 17.353 mg (17.353 mg)	<Comment> 7319-2	<Temp.program [C] [C/min] [min]> 1* 20.0- 550.0 10.00 0.00
<Date> 95/09/19 08:59	<Reference> pt pan 0.000 mg	<Sampling> 0.5 sec	<Gas> nitrogen 300.0 ml/min; 0.0 ml/min



Chemical Process Systems

3-260

WHC-SD-WM-DP-145, REV 1

3-261

DSC

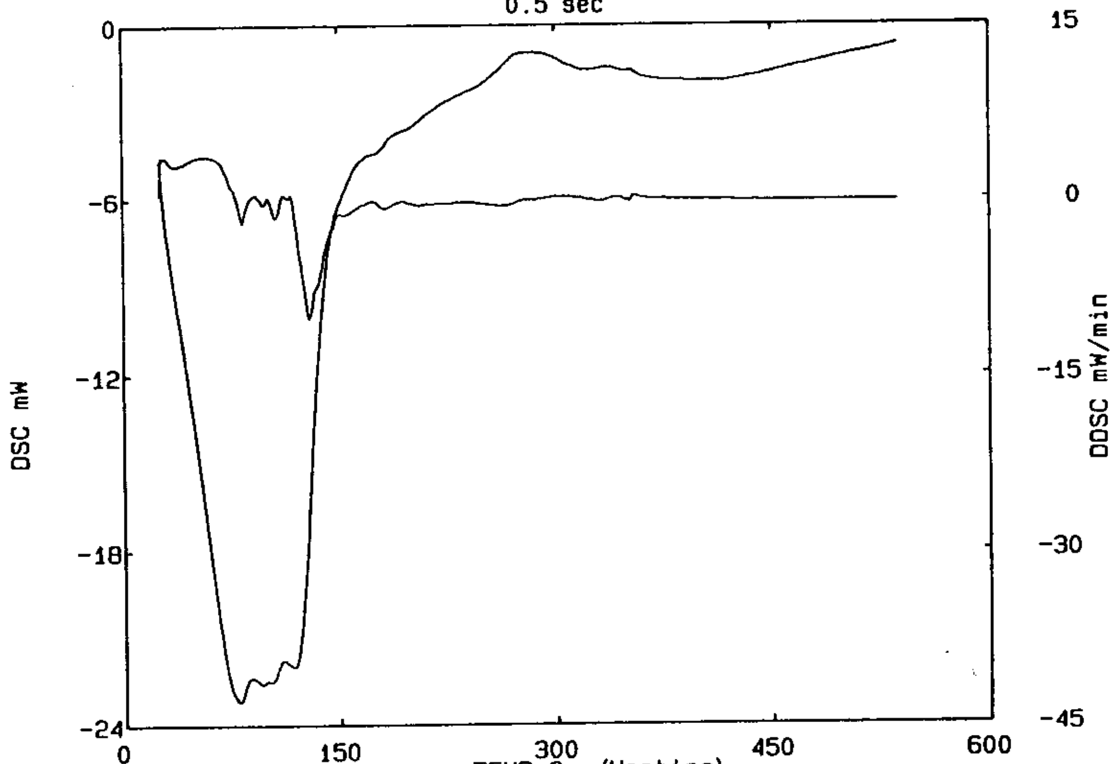
<Name>
18sept95e
<Date>
95/09/18 17:33

<Sample>
18sept95e
15.000 mg
(15.000 mg)
<Reference>
pt pan
0.000 mg

<Comment>
7318

<Sampling>
0.5 sec

<Temp.program [C] [C/min] [min]>
1* 20.0- 550.0 10.00 0.00
<Gas>
nitrogen 300.0 ml/min
--------------- 0.0 ml/min



Chemical Process Systems

WHC-SD-WM-DP-445, REV. 1

1Nn

3-262

TG/DTA

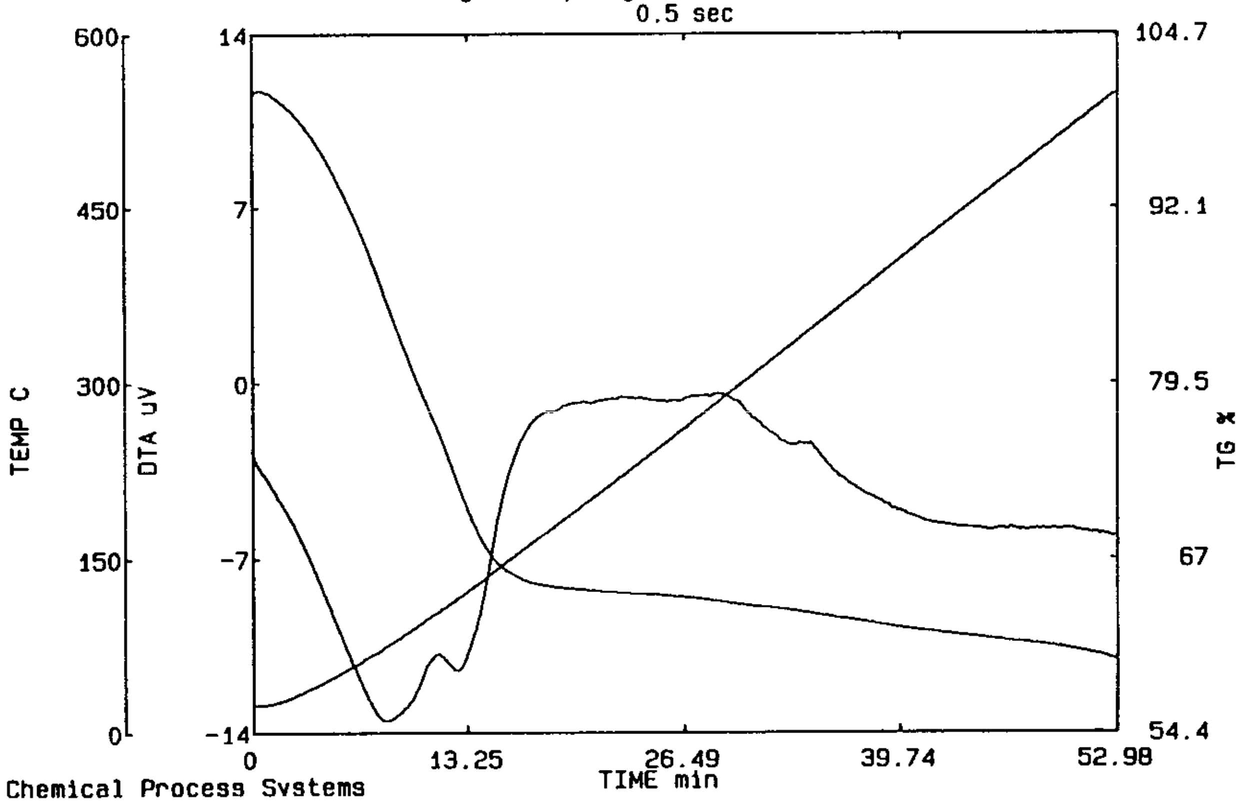
<Name>
18sept95f
<Date>
95/09/18 17:35

<Sample>
18sept95f
11.944 mg
(11.944 mg)
<Reference>
pt pan
0.000 mg

<Comment>
7318

<Sampling>
0.5 sec

<Temp.program [C] [C/min] [min]>
1* 20.0- 550.0 10.00 0.00
<Gas>
nitrogen 300.0 ml/min
0.0 ml/min



Chemical Process Systems

3-263

DSC

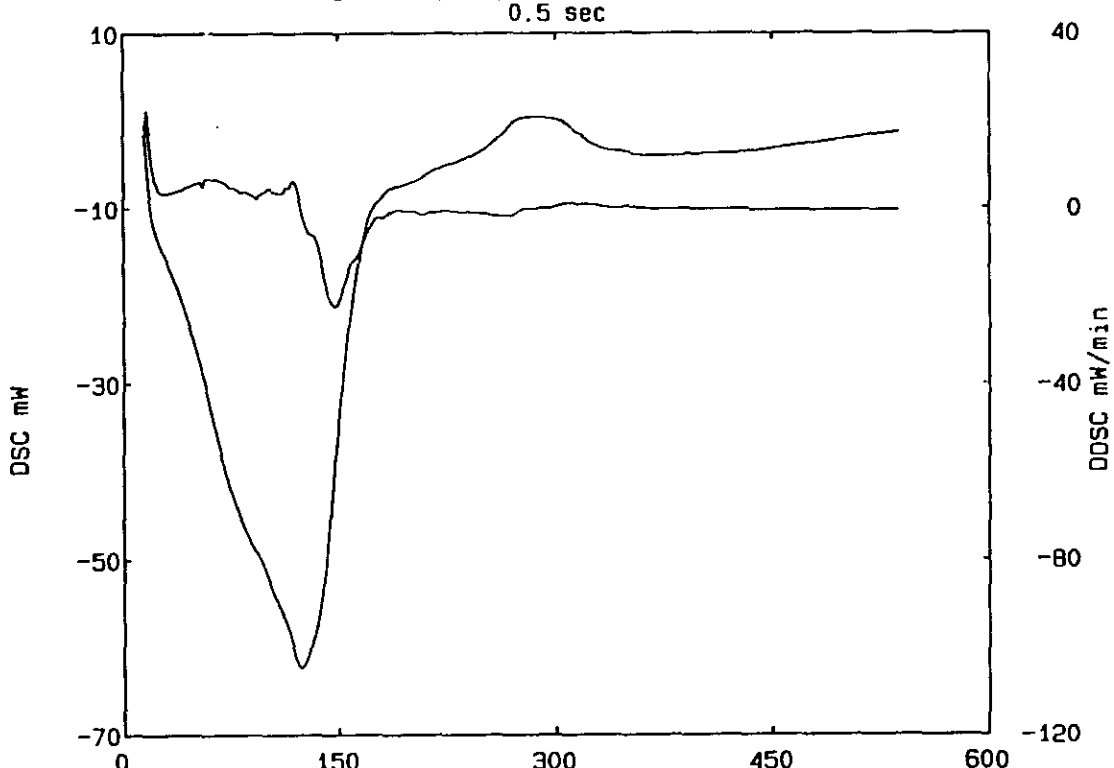
<Name>
18sept95g
<Date>
95/09/18 19:31

<Sample>
18sept95g
36.006 mg
(36.006 mg)
<Reference>
pt pan
0.000 mg

<Comment>
7318-2

<Sampling>
0.5 sec

<Temp.program[C] [C/min] [min]>
1* 20.0- 550.0 10.00 0.00
<Gas>
nitrogen 300.0 ml/min
--------------- 0.0 ml/min



Chemical Process Systems

WHC-SD-WM-DP-145, REV. 1

3-264

TG/DTA

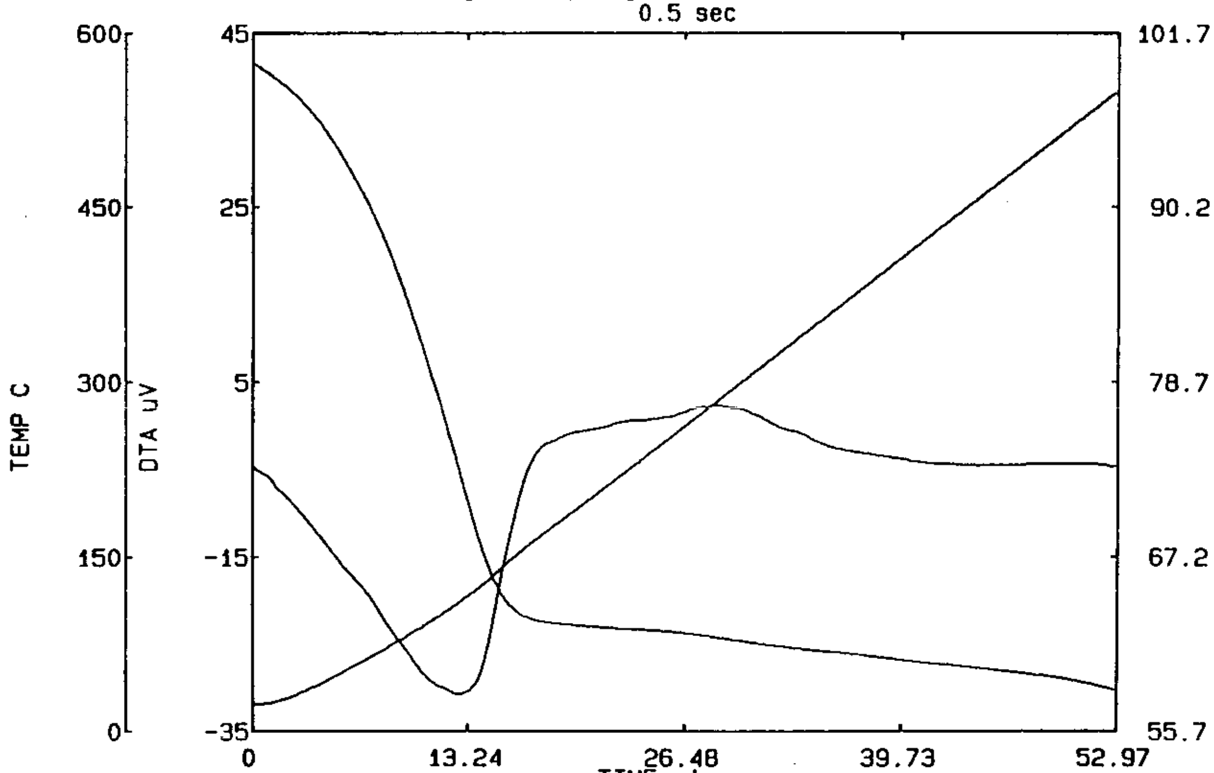
<Name>
18sept95h
<Date>
95/09/18 19:34

<Sample>
18sept95h
26.051 mg
(26.051 mg)
<Reference>
pt pan
0.000 mg

<Comment>
7318-2

<Sampling>
0.5 sec

<Temp.program(C) [C/min] [min]>
1* 20.0- 550.0 10.00 0.00
<Gas>
nitrogen 300.0 ml/min
---------------- 0.0 ml/min



Chemical Process Systems

WHC-SD-WM-DP-145, REV. 1

3-265

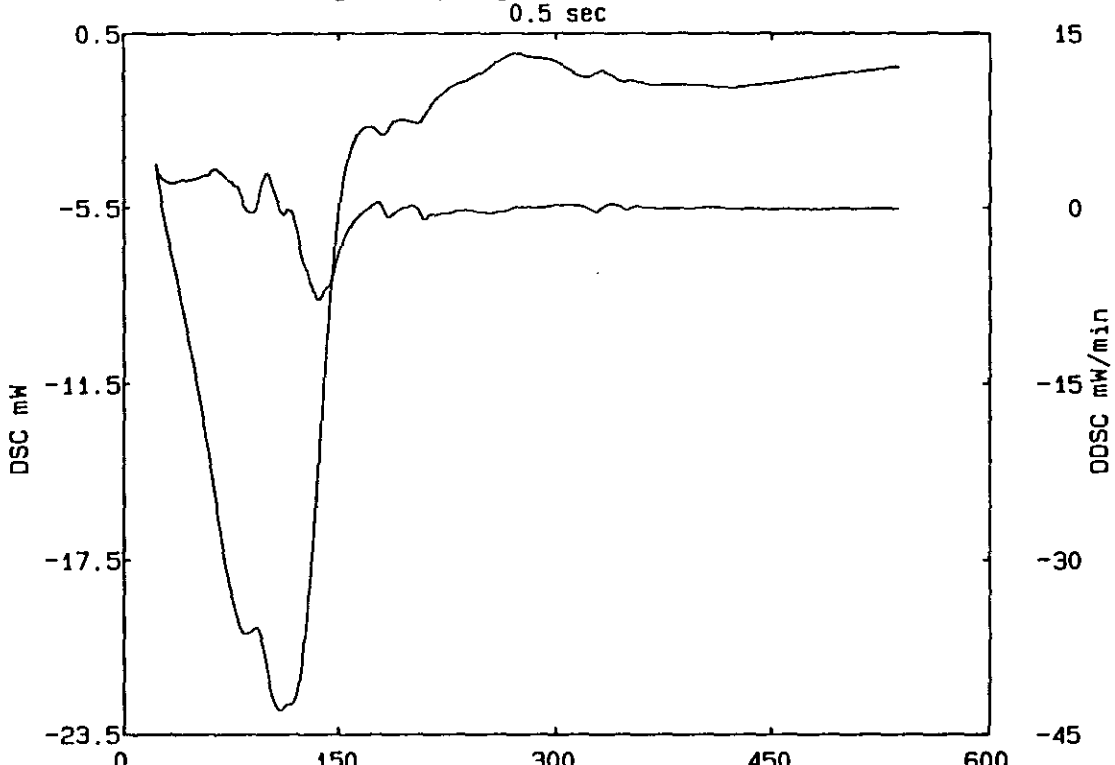
DSC

<Name>
18sept95a
<Date>
95/09/18 09:46

<Sample>
18sept95a
13.397 mg
(13.397 mg)
<Reference>
pt pan
0.000 mg

<Comment>
7317
<Sampling>
0.5 sec

<Temp.program [C] [C/min] [min]>
1* 20.0- 550.0 10.00 0.00
<Gas>
nitrogen 300.0 ml/min
0.0 ml/min



Chemical Process Systems

WHC-SD-WM-DP-145, REV. 1

3-265

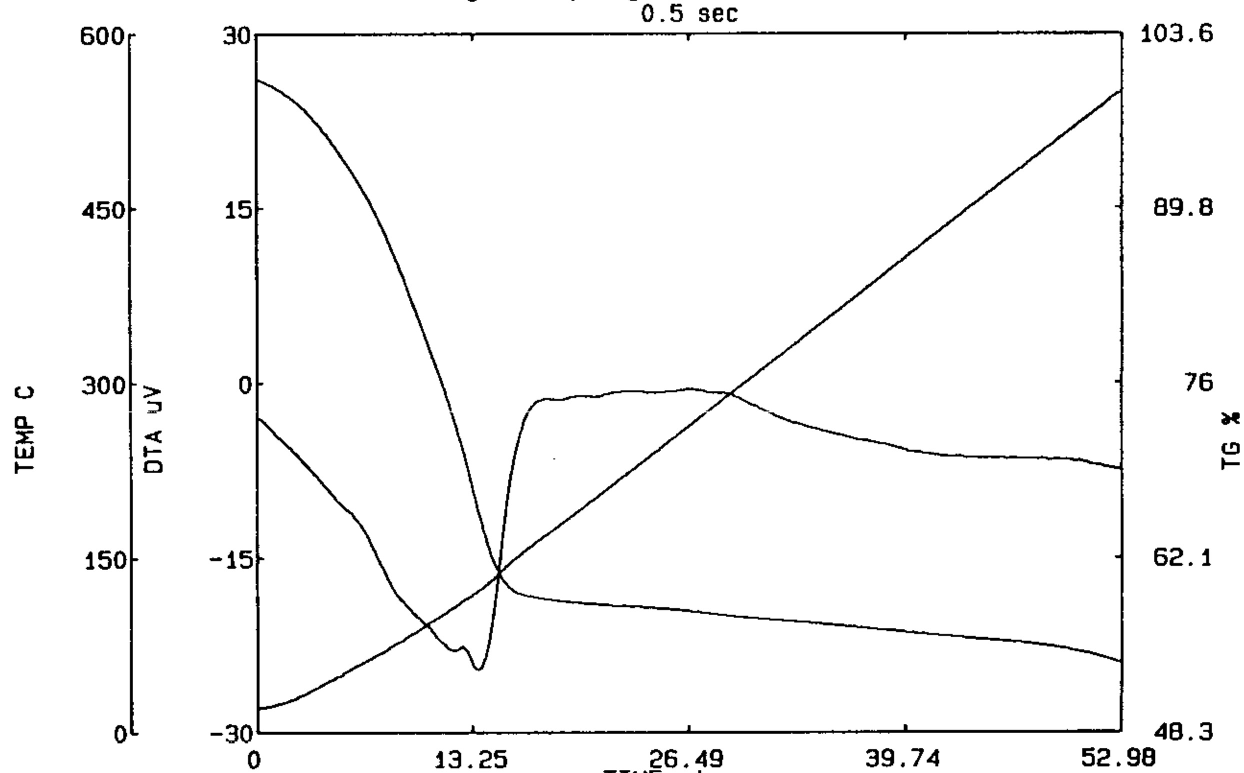
TG/DTA

<Name>
18sept95b
<Date>
95/09/18 09:49

<Sample>
18sept95b
14.483 mg
(14.483 mg)
<Reference>
pt pan
0.000 mg

<Comment>
7317
<Sampling>
0.5 sec

<Temp.program[C] [C/min] [min]>
1* 20.0- 550.0 10.00 0.00
<Gas>
nitrogen 300.0 ml/min
0.0 ml/min



Chemical Process Systems

WHC-SD-WM-DP-145, REV. 1

3-267

DSC

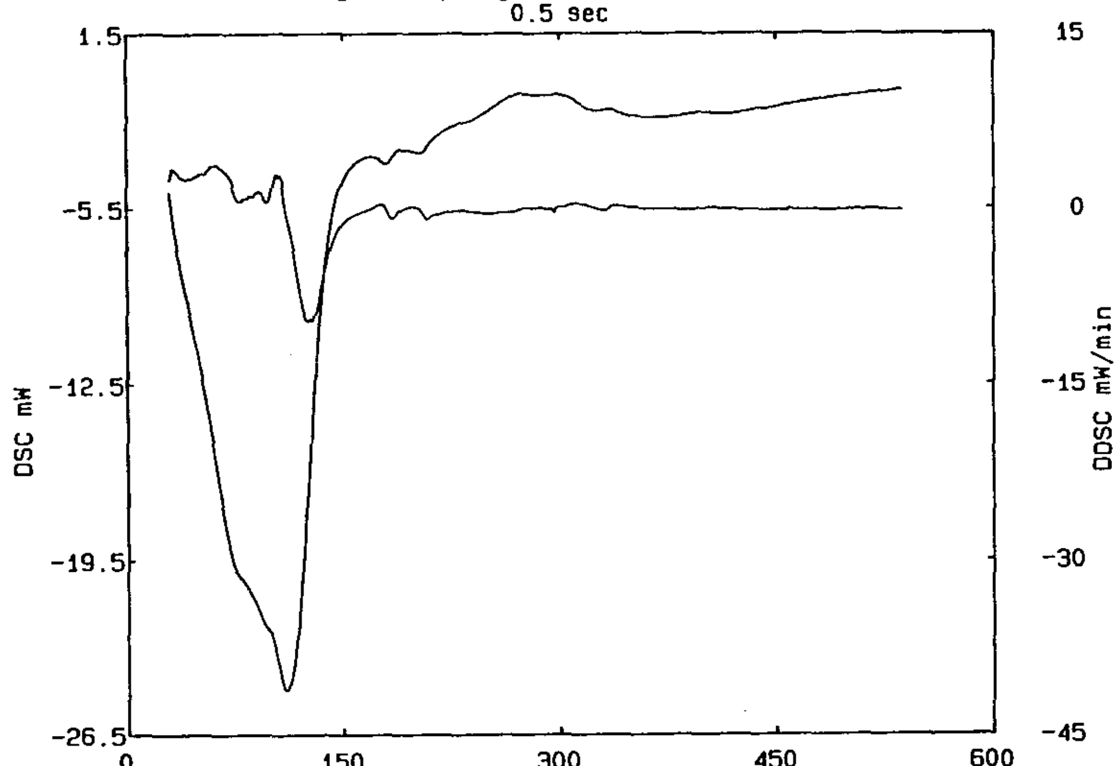
<Name>
18sept95c
<Date>
95/09/18 13:16

<Sample>
18sept95c
13.697 mg
(13.697 mg)
<Reference>
pt pan
0.000 mg

<Comment>
7317-2

<Sampling>
0.5 sec

<Temp.program[C] [C/min] [min]>
1* 20.0- 550.0 10.00 0.00
<Gas>
nitrogen 300.0 ml/min
0.0 ml/min



Chemical Process Systems

WHC-SD-WM-DP-145, REV 1

3-268

TG/DTA

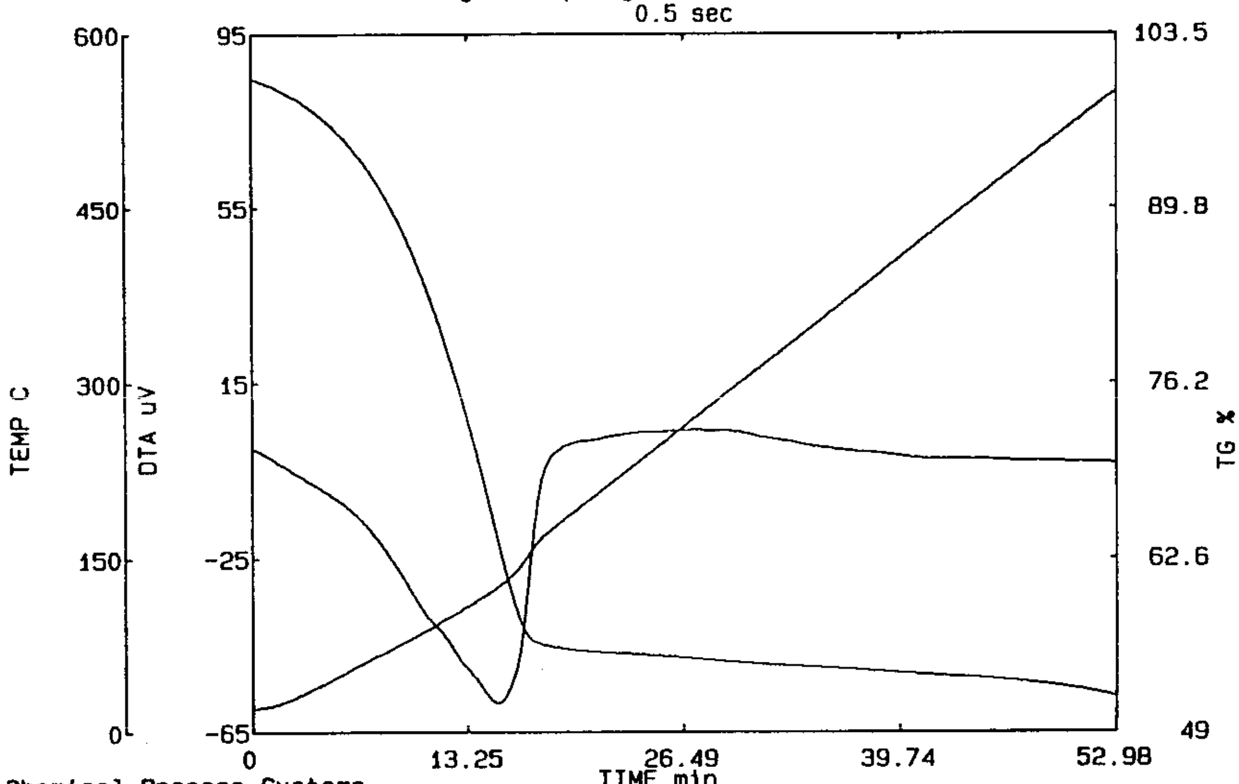
<Name>
18sept95d
<Date>
95/09/18 13:19

<Sample>
18sept95d
36.730 mg
(36.730 mg)
<Reference>
pt pan
0.000 mg

<Comment>
7317-2

<Sampling>
0.5 sec

<Temp.program [C] [C/min] [min]>
1* 20.0- 550.0 10.00 0.00
<Gas>
nitrogen 300.0 ml/min
---------------- 0.0 ml/min



Chemical Process Systems

WHC-SD-WM-DP-145, REV 1

B3 - BULK DENSITY ANALYSIS

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DATE TO QC: 10/09/95 WHC-SD-WM-DP-145, REV. 1

DATA QUALITY REVIEW

I have reviewed the following data for completeness and for compliance with project requirements.

Analyte - BULK DENSITY

Data Package/Report - BY-108 Core 99

Project No. - 21372

ACL Numbers -	7322	7317	7313	7314	7316
	7318	7319	7315	7397	7398

10/9/95

PNL ~~ACL~~ Quality Representative

Date

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WHC-SD-WM-DP-145, REV. 1

Density Measurement

Tank 241-BY-108 Core 99

General Instruction:

1. Keep the sample in a sealed container as much as possible to prevent the sample from drying.
2. All requested weights fro sample plus container are for a closed container.
3. Identify the instrument used for any measurement by reference number or by calibration ID and provide the calibration expiration date.
4. Check the blances daily with the wieghts i the cell. Record the check weights on the test instruction or data sheets.

M&TE List:

C-Cell Balance:

Calib ID 388-06-01-020 Calib. Exp. Date 11/7/95

Signature Scott T[illegible] Date 9/22/95 LRB ________ Page ______

1. Check the balance calibration with a the check weight available in the cell.

   Measured Mass 199.998 g

   Actual Mass 200.050 g

2. Measure the density of each of the solid samples using the displacement method. Mineral Oil should be used as the fluid. Graduated cylinders should be used to determine the volume of the mineral oil and the sample. Record these volumes and masses.

Sample #7322

	mass		Volume	
oil	21.374 g	oil	3.20 ml	
oil + sample	21.6071 g	oil + sample	3.35 ml	
sample	0.227 ~~0.233~~ g gT 9/28/95	sample	0.15 ml	

description peanut butter type sludge

density = sample mass/sample volume= 0.227 gT 9/28/95 ~~0.233~~ g/ 0.15 ml= 1.51 ~~1.55~~ g/ml gT 9/28/95

Sample #7317

	mass		Volume
oil	22.193 g	oil	4.15 ml
oil + sample	22.674 g	oil + sample	4.46 ml
sample	0.481 g	sample	0.31 ml

description peanut butter type Sludge

density = sample mass/sample volume= 0.481 g/ 0.31 ml= 1.55 g/ml

Signature Scott T [signature] Date 9/22/95 LRB ________ Page ______

Sample #7313

	mass			Volume	
oil	21.631	g	oil	3.80	ml
oil + sample	21.950	g	oil + sample	4.00	ml
sample	0.319	g	sample	0.20	ml

description hard, dry solids

density = sample mass/sample volume= 0.319 g/ 0.20 ml= 1.60 g/ml

Sample #7314

	mass			Volume	
oil	21.950	g	oil	4.00	ml
oil + sample	22.370	g	oil + sample	4.31	ml
sample	0.420	g	sample	0.31	ml

description almost liquid like

density = sample mass/sample volume= 0.420 g/ 0.31 ml= 1.35 g/ml

Sample #7316

	mass			Volume	
oil	20.920	g	oil	3.00	ml
oil + sample	21.234	g	oil + sample	3.20	ml
sample	0.314	g	sample	0.20	ml

description hard, dry Solids

density = sample mass/sample volume= 0.314 g/ 0.20 ml= 1.57 g/ml

Signature Scott T[illegible] Date 9/22/95 LRB ________ Page ______

Sample #7318

	mass			Volume	
oil	20.367	g	oil	2.30	ml
oil + sample	20.520	g	oil + sample	2.40	ml
sample	0.153	g	sample	0.10	ml

description peanut butter

density = sample mass/sample volume= 0.153 g/ 0.10 ml= 1.53 g/ml

Sample #7319

gT 9/22/95

	mass			Volume	
oil	21.243 ~~20.532~~	g	oil	3.35	ml
oil + sample	21.546 ~~20.759~~	g	oil + sample	3.55	ml
sample	0.303 ~~0.227~~	g	sample	0.20	ml

description peanut butter like sludge

density = sample mass/sample volume= 0.303 g/ 0.20 ml= 1.52 g/ml

Sample #7315

	mass			Volume	
oil		g	oil		ml
oil + sample		g	oil + sample		ml
sample		g	sample		ml

not enough sample to determine density gT 9/22/95

description small balls of solid material (tapioca pudding)

density = sample mass/sample volume= g/ ml= g/ml

Signature Scott T[illegible] Date 9/22/95 LRB ______ Page ______

3. Measure the density of each of the drainable liquid samples using the a fixed pipet to deliver an accurate volume and measuring the mass of the sample with a balance. Record these volumes and masses.

Sample # 7397

sample volume 15.0 µL
sample mass 20.316 mg
density = sample mass/sample volume= 20.316 mg/ 15.0 µL= 1.35 g/ml

sample volume 25.0 µL
sample mass 30.332 mg
density = sample mass/sample volume= 30.332 mg/ 25.0 µL= 1.21 g/ml

sample volume 15.0 µL
sample mass 18.083 mg
density = sample mass/sample volume= 18.083 mg/ 15.0 µL= 1.21 g/ml

sample volume 25.0 µL
sample mass 30.332 mg
density = sample mass/sample volume= 30.332 mg/ 25.0 µL= 1.21 g/ml

description clear liquid with small amount of settled solids

Signature Scott T[illegible] Date 9/22/95 LRB ________ Page ______

Sample # 7398

sample volume 20.0 µL
sample mass 26.977 mg
density = sample mass/sample volume= 26.977 mg/ 20.0 µL= 1.35 g/ml

sample volume 20.000 µL
sample mass 24.205 mg
density = sample mass/sample volume= 24.205 mg/ 20.000 µL= 1.21 g/ml

sample volume 25.000 µL
sample mass 29.784 mg
density = sample mass/sample volume= 29.784 mg/ 25.0 µL= 1.19 g/ml

sample volume 15.0 µL
sample mass 18.818 mg
density = sample mass/sample volume= 18.818 mg/ 15.0 µL= 1.25 g/ml

description clear liquid with a small amount of settled solids

Signature Scott [signature] Date 2/22/95 LRB ________ Page ______